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The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

Volume 73 Number 6

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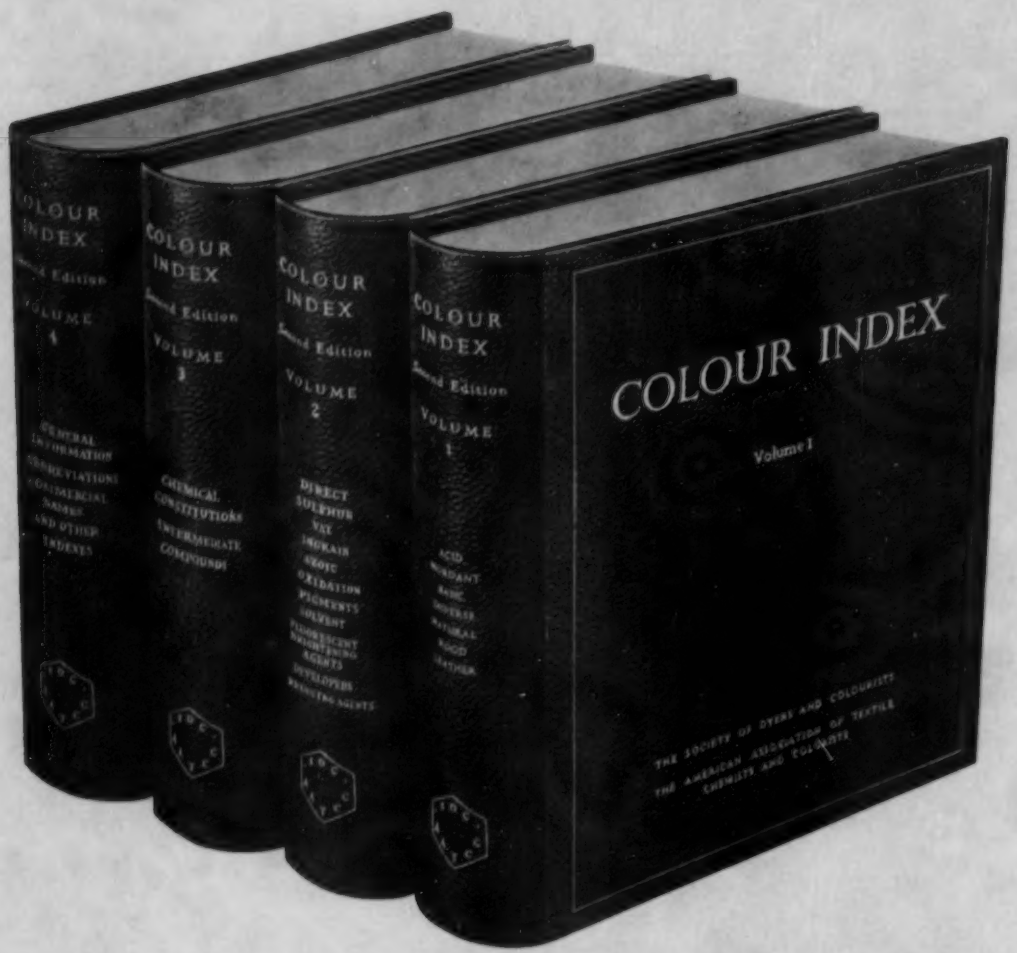

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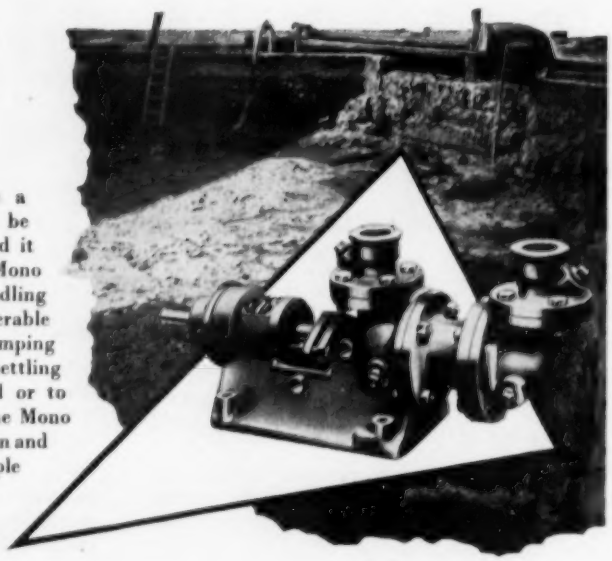

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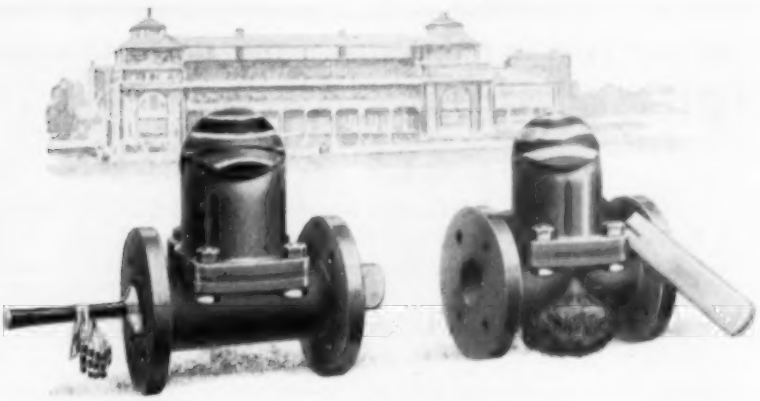

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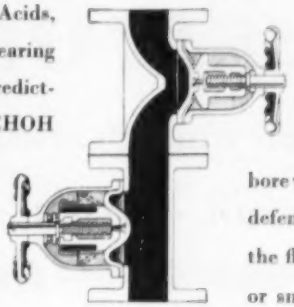

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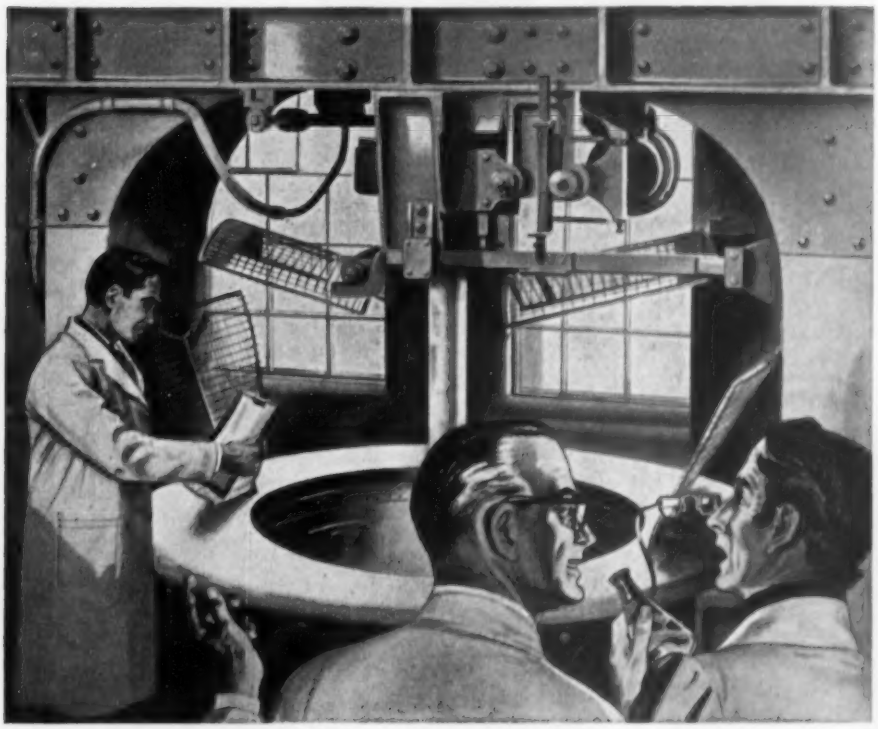

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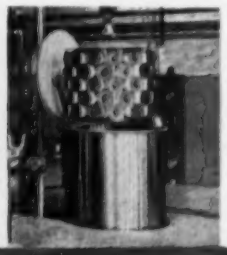

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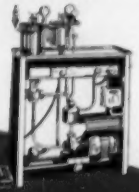

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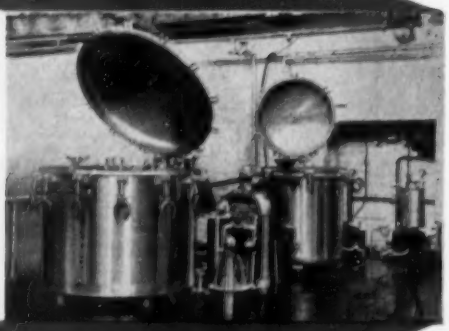

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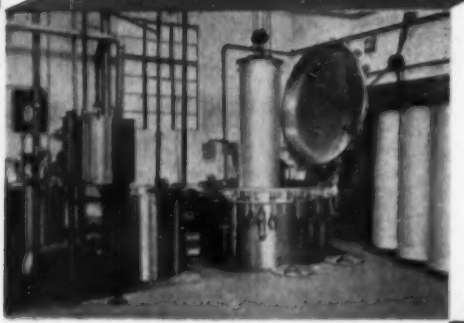

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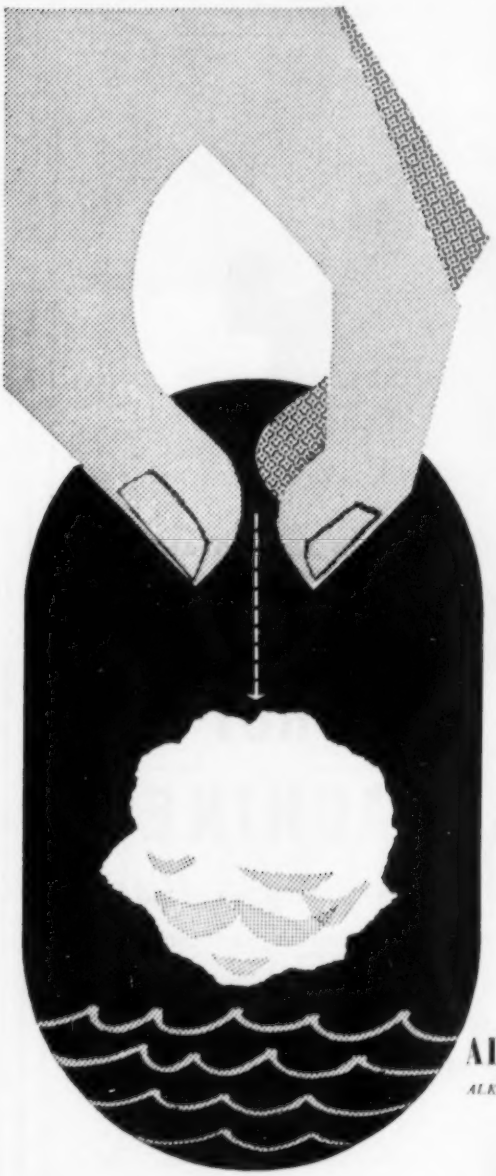

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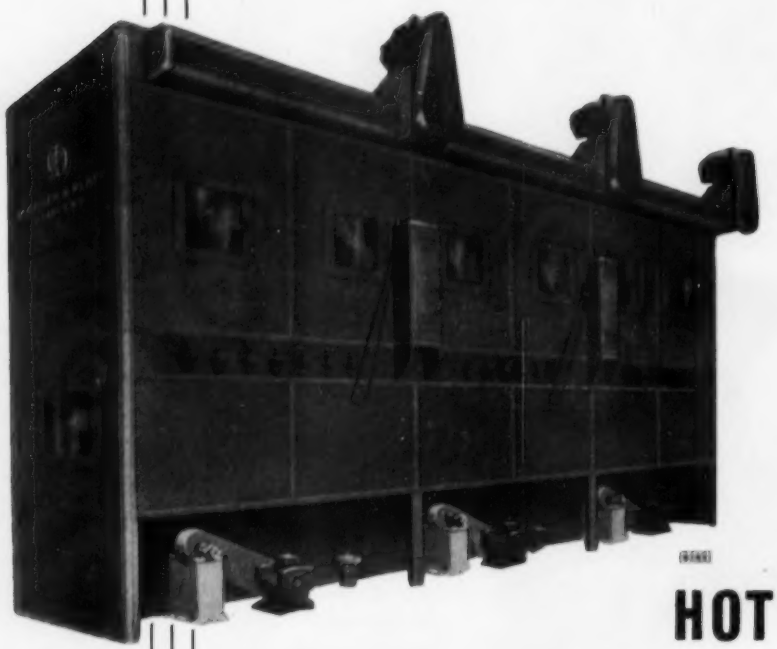

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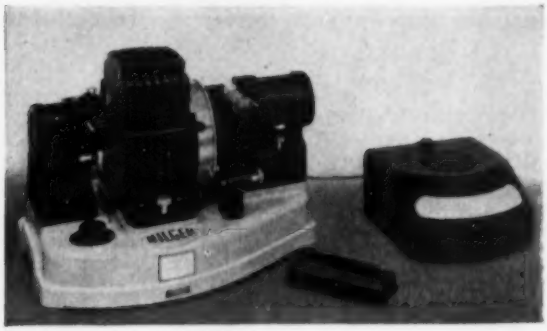

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Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–7 of the January 1957 and pages 313–317 of the July 1956 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138–40). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

LECTURES

A New Approach to the Continuous Dyeing of Secondary Cellulose Acetate, Cellulose Triacetate, and Terylene Polyester Fibre — the Vapocol Process — *D. A. Garrett*

Dyeing (George Douglas Lecture) — *G. S. J. White*

COMMUNICATIONS

Relationship between Physical State and Rate of Fading of Dyes — *G. Baxter, C. H. Giles, and W. J. Lewington*

Determination of the Migratory Properties of Direct Dyes — *J. Cegarra*

The Colour Works of Perkin & Sons—
Some Hitherto Unrecorded Details
Pacta Conventa — The Last Days of Perkin & Sons
Litera Scripta Manet — The Alizarin Débâcle — *W. H. Cliffe*

The Variation of Colour Fastness on Fabrics — *S. Horrdin and Ulf Bülow*

The Use of Paper Chromatography in the Recognition of Chemical Damage to Wool — *V. Köpke and B. Nilssen*

The Measurement of Donnan Potentials with Dyed Cellulose and Aqueous Salt Solutions — *S. M. Neale and P. K. Saha*

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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 73 Number 6 JUNE 1957 *Issued Monthly*

Proceedings of the Society

Reactive Dyes for Textiles

T. VICKERSTAFF

Meetings of the London Section held in the Rooms of the Royal Society, Burlington House, on 5th October 1956, Dr. H. W. Ellis in the chair; of the Manchester Section held at the Textile Institute on 19th October 1956, Mr. J. Boulton in the chair; of the Midlands Section held at the Albert Hall Institute, Nottingham, on 31st October 1956, Mr. C. A. Mills in the chair; of the Huddersfield Section held at Silvio's Cafe on 20th November 1956, Mr. J. Calvert in the chair; and of the West Riding Section held at the Victoria Hotel, Bradford, on 6th December 1956, Mr. C. W. Green in the chair

Of various methods of achieving wet fastness in dyeings, direct chemical linkage of the dye to the fibre has not hitherto been employed in practice. The Procion dyes are a new class of dyes, which will react with cellulose in presence of alkali. The evidence for chemical reaction is reviewed. Being dyes of small molecular weight, their affinity for cellulose is low and diffusion inside cellulose fibres rapid, so that cold dyeing becomes feasible. Also, side-reaction of the dyes with water is minimised at low temperatures. The importance of these properties in determining the preferred dyeing methods is discussed.

Procion dyes can be applied in practice by continuous processes, including a method which entails only padding and drying. Novel batch dyeing methods have been developed. The dyes can also be applied by printing with a suitable thickener, and again a novel non-steaming printing style has been evolved and a particularly interesting style using white and vat-dye-illuminated resists under Procion dyes.

The fastness properties characteristic of chemical linkage are briefly reviewed.

INTRODUCTION

One of the major problems facing the dyer from time immemorial has been the production of coloured fabrics possessing good fastness to water and washing. In essence this involves attaching a dye molecule to a fibre substance in such a way that it cannot be easily removed. There are only three possible ways of attaching dyes to fibres— (*a*) by physical adsorption, (*b*) by mechanical retention, and (*c*) by chemical reaction. All these possibilities have been explored by dye chemists, but up to the present day only the first two have been found of real value, and they form the basis of almost all existing dyeing methods (solution of dye in the fibre might be regarded for this purpose as a physical process, but the dyeing of protein fibres with metal-complex dyes is an example of chemical reaction, with the metal atom chelating both fibre and dye).

Physical adsorption is the basis of direct dyeing on cellulose fibres and acid dyeing on wool and is in many ways very satisfactory. So long as water-soluble dyes are employed, however, dyeing must be a reversible equilibrium between adsorbed dye and dye in aqueous solution, and consequently it is theoretically impossible with such a system to obtain the very highest degree of wet fastness. Wet fastness can be increased by altering the shape of the dye molecule to increase the adsorption forces, but such modifications usually reduce water-solubility and increase the tendency of the dye to aggregate, and these properties combined with the higher affinity make the production of level dyeings more difficult.

Mechanical retention forms the basis of vat, sulphur, and azoic dyeing and of the mass colouring of man-made fibres. High wet fastness is achieved through the virtual insolubility of the pigment in water or soap solution, together with the mechanical retention of the pigment particles by the fibre substance in which they are embedded. With this mechanism, satisfactory wet fastness can be obtained, but the process of introducing the insoluble pigment into the fibre is complicated and troublesome, and occasional difficulty may arise from growth of pigment particles or deposits of loose dye particles on the surface of the fibres.

Thus both existing methods of attaching dyes to fibres have their disadvantages, and interest in the third possible mechanism— chemical reaction— is of long standing. This interest arises from the general supposition that, if a dye could be attached to a fibre by a covalent link, it should become part of the fibre, and, therefore, should have high wet-fastness properties. Perhaps this view exaggerates the stability of chemical linkages, as an ester link, for example, might hydrolyse fairly rapidly under alkaline soap boiling conditions. Rubbing fastness with chemically linked dyes should present no problem, for the dye could be rubbed away only by removal of the fibre substance itself.

Such considerations form the justification for attempting to link dyes chemically to fibres. One of the earliest attempts in the case of cellulose fibres was that of Cross and Bevan [1], who in 1895 carried out the following series of reactions—

Cellulose
↓ + Caustic soda
Soda cellulose
↓ + Benzoyl chloride
Benzoylcellulose
↓ + Nitric acid
Nitrobenzoylcellulose
↓ + Ammonium sulphide
Aminobenzoylcellulose
↓ Diazotisation
Diazobenzoylcellulose
↓ Couple with phenolic compounds
Arylazobenzoylcelluloses

Regarding the last stages in this process, Cross and Bevan state: ". . cellulose amidobenzoate was diazotised and combined with amines and phenols to form red and yellow colouring matters, the reacting residue remaining more or less firmly combined with the cellulose."

This process was, of course, merely of academic interest in the study of cellulose structure, but many attempts were made subsequently to simplify the process to something of practical value. Cellulose itself or soda cellulose was treated with acid chlorides, alkyl halides, heterocyclic halides, chloroacetophenone, etc. [2] to yield derivatives which could react further with dyes or intermediates to give coloured fibres. None of the processes was of practical value because of the severity of the reactions, which degraded the fibre, and because of the general need to use at some stage expensive non-aqueous reaction media with all their attendant difficulties.

The reason for this complicated approach to the problem was the belief that the hydroxyl groups of cellulose were relatively inert, so that drastic conditions were needed to promote reaction. The alternative approach of using a highly reactive grouping in the coloured molecule was similarly considered impracticable, unless, again, a non-aqueous system could be used, since a grouping highly reactive towards hydroxyl groups would probably be hydrolysed by water. On this theme attempts have been made to develop a practicable single-stage process by reaction, for example, of quaternary bases with cellulose under dry baking conditions [3], while recently it has been shown that certain sulphonic esters of dyes will react with cellulose at high temperatures [4].

Naturally, the search for more practical methods of using reactive dyes has continued, and this work has now culminated in the production of a new range of dyes— the Procion (ICI) dyes— which are water-soluble but contain a reactive group capable of combining with cellulose under alkaline conditions. The dyes are not so highly reactive as to be unstable in water at room temperatures, but yet they are sufficiently reactive to avoid the need for drastic fixation conditions. Reaction will occur in presence of sodium carbonate solution even in the cold, although stronger alkalis can be used if desired. Reaction with cellulose under such mild conditions seems so contrary to previous views that it is desirable to review the evidence that a chemical reaction between dye and fibre does take place.

EVIDENCE FOR REACTION OF PROCION DYES WITH CELLULOSE

The occurrence of a chemical reaction between Procion dyes and cellulose is indicated by the following facts—

(1) Procion dyes are highly soluble in water, and the solubility is conferred on the molecule by the presence of sulpho ($-SO_3H$) groups. There can be no question, therefore, of a temporary solubilisation phenomenon. If cotton is padded or printed with a neutral solution of a Procion dye and then dried, the colour can be removed almost completely from the fabric by water washing. If, however, the impregnated fabric is treated with alkali, a large proportion of the dye is fixed and is resistant to soap boiling. This can be demonstrated very simply by cutting a printed but unfixed fabric into two parts, immersing one half in sodium bicarbonate solution, and then ironing the fabric. When boiled in soap solution, the alkali-treated fabric retains its colour, whereas the dye is washed out of the untreated fabric. Since the dye molecule still contains its water-solubilising groups, this behaviour can be explained only by reaction with the fibre.

(2) Cotton dyed with direct, vat, or azoic dyes can be stripped by boiling with a solvent such as pyridine, *o*-chlorophenol, or chloroform. These solvents do not extract Procion dyes from dyed cellulose, which suggests that some force other than adsorption or mechanical retention is holding the dye on the fibre.

(3) Polyvinyl alcohol may be regarded as similar in nature to cellulose from the point of view of reactivity. If aqueous solutions of polyvinyl alcohol, containing on the one hand a Procion dye and on the other an acid or a direct dye, are poured on to the surface of a solution of caustic soda saturated with salt, a film is formed. The film containing the Procion dye can subsequently be boiled in water and will not disintegrate, although it swells somewhat, whereas the film containing an acid dye rapidly dissolves.

(4) In a similar manner, cellulose which has been dyed with Procion dyes under certain conditions swells but does not dissolve in cuprammonium solution, whereas direct-dyed cotton dissolves rapidly. This fact and the behaviour of the polyvinyl alcohol film suggest very strongly that reaction with the hydroxyl groups of the polymer has occurred to change its solubility.

(5) The colour of Procion Yellow R is due to the presence of an azo group. Consequently this dye can be reduced on the fibre by sodium hydrosulphite to two amine-containing products. If the dye has reacted with the fibre before reduction, one of these products should remain anchored to the fibre and should resist washing out. Such a "discharged" fibre can in fact be diazotised and coupled with another phenol or amine to produce a new dye, and this process can be repeated indefinitely. Again, this is very strong evidence that the Procion dye is combined with the fibre.

Considering all these points together, it may be concluded that the occurrence of chemical reaction in the dyeing of cellulose with Procion dyes is well established.

THE DYEING PROPERTIES OF REACTIVE DYES

A satisfactory process of dyeing with any reactive dye must consist of two stages— entry of dye into the fibre and chemical reaction within the fibre. It is possible to devise dyeing methods in which both processes are going on at the same time, and these will be discussed later, but in order to get the clearest picture of the mechanism of the process, it is best to study the two stages separately. In the case of Procion dyes this is comparatively easy, for reaction occurs only in the presence of alkali. Hence it is possible to investigate first the application of the dyes in neutral solution, when they behave as non-reactive dyes, and then to study the reactions which occur when alkali is added.

The most interesting feature of a reactive system is, of course, the actual process of combination between dye and cellulose. The structure of a Procion dye may be represented as S–R–X, where S represents one or more sulpho groups conferring solubility on the molecule, R is the coloured organic molecule, and X is a reactive halogen atom. As already mentioned, it is difficult to conceive of any system which will react with the hydroxyl groups of cellulose and yet will not react with water. It may be anticipated, therefore, that in the fixation process two reactions will proceed simultaneously—

$$\text{S·R·X} + \text{HO·Cell} \rightarrow \text{S·R·O·Cell} + \text{HX} \qquad \text{(i)}$$

$$\text{S·R·X} + \text{HOH} \rightarrow \text{S·R·OH} + \text{HX} \qquad \text{(ii)}$$

Reaction (i) is the desired reaction leading to fixation, while (ii) is the side-reaction leading to the formation of an inactive hydroxy derivative of the original dye. This dye will still be water-soluble, as the sulpho groups are not affected by the reaction, but it must be removed if the dyed fibre is to attain the full wet fastness characteristic of the covalently linked dye. It will be convenient in subsequent discussion to refer to the original reactive dye (S·R·X) as *reactive dye*, the dye combined with cellulose as *bound dye*, and the hydroxy derivative (S·R·OH) as *loose dye*.

At first sight there seems no reason why reaction of the dye with cellulose should take place preferentially, but in practice the degree of fixation attained usually exceeds 70% of the dye present in the system under recommended conditions of application. Above that figure the actual fixation depends on the particular dye employed and upon the temperature, the pH, and other constituents of the dye liquor at the time of fixation. In general, high temperatures and strong alkalis favour the side-reaction and lead to lower fixation. Under no conditions so far examined has the occurrence of the side-reaction been entirely prevented.

A possible explanation for the preferential reaction with cellulose is that reaction can proceed more easily when the dye molecule is adsorbed on a cellulose surface. To confirm this hypothesis attempts have been made to measure the actual rates of reaction of the dyes with water and with cellulose. Cellulosic material which has been dyed from neutral solution with Procion dyes has been plunged into alkaline solution, and then after varying times transferred to acid solution to stop the reaction. It has not been found possible to obtain a definite estimate of the rate, as the reaction is complete in less than 5 sec. even at room temperature. It may be concluded, therefore, that for practical purposes the reaction of adsorbed dye with cellulose is instantaneous, and that overall rates of fixation in practice will be determined by the rates of diffusion of the dyes into the fibre or by the rate of diffusion of hydroxyl ions into the fibre.

The reaction of Procion dyes with water has been followed by calorimetric determination of the heat evolved in the process and is much slower: Procion Brilliant Red 2B, for example, has a time of half decomposition around 20 min. at room temperature. Details of the reaction rate at different temperatures are shown in Table I, and the comparative rates of reaction with cellulose and with water in Fig. 1.

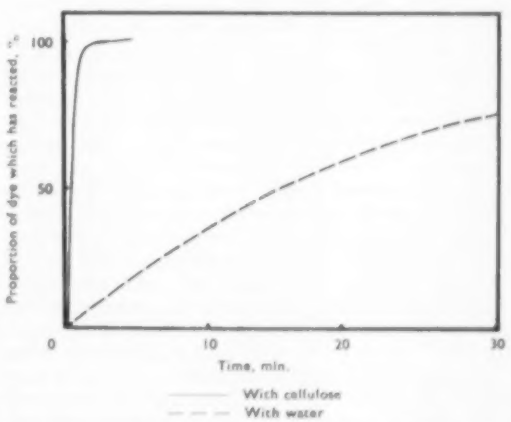


FIG. 1— Relative Speeds of Reaction of Procion Dyes with Water and with Cellulose

TABLE I

Hydrolysis of Procion Brilliant Red 2BS in Solution

(0·33 g. dye (1%) + 30 g. salt + 10 g. sodium carbonate per litre; pH = 11·1)

Temp. (°C.)	k (min.$^{-1}$)	$t_{\frac{1}{2}}$ (min.)
25	0·05	13·8
30	0·09	7·7
35	0·13	5·3

Activation energy = 22,600 cal./mole

These facts have a very important bearing on practical dyeing methods. If the reactive dye is distributed between a fibre and a neutral dyebath with a large part of the dye still remaining in solution, then when alkali is added reaction will commence both in the fibre and in solution. In the fibre at any temperature reaction will be extremely rapid, and the bound dye will be removed from the equilibrium which previously existed between the reactive dye in the fibre and that in the bath. Hence more of the reactive dye can diffuse into the fibre to be fixed in its turn. The extent to which this can occur will depend on the relative rate at which the dye can diffuse into the fibre compared with the rate at which it is being inactivated in the dyebath. Fortunately, as will be noted later (p. 241), the rate of diffusion of these dyes is high, so that a favourable result can be obtained. Both rates will increase with increasing temperature. Observations at different temperatures show that the rate of decomposition increases more rapidly than the rate of diffusion, so that at higher temperatures there is less chance of additional dye diffusing into the fibre before it is decomposed in solution. Consequently, in carrying out fixation of the Procion dyes in conventional dyebaths it is preferable to work at relatively low temperatures in order to get maximum fixation. This does not, of course, apply to processes involving padding, where most of the dye is contained within the fibre to begin with and the question of diffusion does not arise.

Considering now the actual method of application of dyes, the dyeing properties of any dye are determined by its affinity and diffusion coefficient. Most existing dyes possess appreciable affinity for the fibre on which they are to be dyed, but in the case of reactive dyes this need not be so. In other words, the affinity of the original reactive dye for the fibre has no direct bearing on the fastness properties of the chemically combined dye, but will profoundly affect the dyeing method.

An unexpected factor in this connection is the influence of the side-reaction with water. With a dye of very low affinity the side-reaction will lead to a hydroxy dye also of low affinity, which will be removed readily from the fibre and, still more important, will not stain adjacent cellulose fibres. On the other hand, if the dye has high affinity, the hydroxy by-product will almost certainly have high affinity also, will be difficult to wash out from the dyed fabric, and if not completely removed will be liable to cause staining of adjacent cellulosic fibres in domestic washing.

As regards dyeing properties, a dye of high affinity would be particularly useful for batch processing, since it could be applied in a controlled way in neutral solution by the addition of salt in the same way as for a direct dye. In this way the greater proportion of the dye could be applied uniformly throughout a package of yarn or a length of fabric before alkali was added to fix the dye *in situ*. Naturally, if the affinity were too high difficulties could arise from unevenness and poor penetration, which the poor migration properties would not overcome. Such a dye would not be very suitable for continuous processing, since the high affinity would lead to tailing.

On the other hand, a dye of low affinity would be suitable for application by continuous processes, as it would give no tailing, but trouble might arise during drying owing to the migration of dye with the evaporating water. A dye of zero affinity could not be applied by batchwise dyeing processes at all, but if the affinity were low but significant, a limited exhaustion could be obtained before reaction, and in this stage the dye should show excellent migration and penetration properties. Difficulties might be expected to arise when alkali was added to fix the dye, as a large proportion would still be in solution, and if this did combine with the fibre it would be fixed wherever it first reached the fibre, for no migration after fixation is theoretically or practically possible. Hence precautions would have to be taken to ensure slow and uniform addition of alkali to obtain a level final result.

TABLE II

Affinities of Dyes for Viscose Rayon at 30°C.

Procion Yellow RS	...	−4,100 cal.
Procion Brilliant Red 2BS		−2,500 cal.
Procion Blue 3GS	...	−2,400 cal.
Chrysophenine GS	...	−4,700 cal.*
Chlorazol Sky Blue FFS	...	−7,500 cal.*

*Extrapolated from data at 60° and 100°C.

In fact, the existing Procion dyes are of the low-affinity type, although there are differences between the individual dyes. Exact determinations of affinity are difficult, as prolonged dyeings to equilibrium cannot be carried out without some decomposition in the dyebath or reaction with the fibre. Approximate affinity measurements at room temperature over 1 hr. give the results in Table II, in which the values for two typical direct dyes are also included. It must be emphasised that these measurements refer to the reactive dye in neutral solution under conditions such that no chemical reaction occurs. The affinity of the bound dye will be extremely high and cannot be determined by a conventional dyeing technique. From the above results it will be apparent that the affinities of the Procion Red and Blue are very low, being only about half that of Chrysophenine G, which itself is a direct dye of very low affinity, while the Procion Yellow has an affinity comparable with that of Chrysophenine G.

To illustrate in a practical manner the significance of these figures, the percentage exhaustions on viscose rayon of these dyes in a

TABLE III

Exhaustions (%) of Procion and Direct Dyes on Viscose Rayon

(30 : 1 liquor ratio)

Temperature ...	20°c.		60°c.	
Salt concn., g/litre	5	30	5	30
Chrysophenine GS ...	93	99	79	82
Chlorazol Sky Blue FFS...	99	100	96	100
Procion Yellow RS ...	85	91	72	75
Procion Brilliant Red 2BS	23	30	9	10
Procion Blue 3GS ...	30	36	11	12

30 : 1 liquor containing 5 and 30 g. of common salt per litre at 20° and 60°c. have been calculated and are shown in Table III. The figures are of doubtful accuracy owing to the uncertainty in the original affinity measurements on the Procion dyes, but they do indicate the practical behaviour which may be expected from these dyes. Clearly, the exhaustion will be very low in presence of the usual additions of salt, but it can be increased by further increase in the salt concentration. The Yellow is very much more salt-sensitive than the Red and the Blue. The table also shows that the exhaustions of these dyes fall considerably with increase of temperature, an effect which is more marked than with direct dyes. To obtain adequate exhaustion of Procion dyes in batchwise dyeing processes, therefore, dyeing has to be carried out at as low a temperature as possible in presence of a high concentration of salt.

The second major characteristic of any dye is its diffusion coefficient into the fibre. The rate of diffusion of a dye inside cellulose depends partly on the size and the shape of the molecule, as in free diffusion in solution, but also to a much greater extent on its affinity for the fibre. The reason for this is that diffusion is a stepwise process from one adsorption site to another, and the ease with which the dye is displaced from a site is, of course, dependent on its affinity. Hence it may be expected that a dye with low affinity will diffuse quickly within the fibre.

TABLE IV

Diffusion Coefficients of Procion Dyes and Direct Dyes in Viscose Film

(30°c. and 5 g. NaCl per litre)

	Diffusion Coefficient (cm.²/sec. × 10^6)
Procion Yellow RS	2·4
Procion Brilliant Red 2BS ...	25·0
Procion Blue 3GS	20·0
Chrysophenine GS	4·1
Chlorazol Sky Blue FFS ...	1·5

The Procion dyes show just this behaviour. The diffusion coefficients of the dyes in Cellophane are compared with those of typical direct dyes in Table IV. It will be seen that the Procion Red and Blue diffuse about twenty times as rapidly as Chlorazol Sky Blue FF. The practical implications of these results are very important and lead to the expectation that Procion dyes will have excellent penetration into fibres and fabrics, excellent levelling (before fixation), and a very rapid rate of dyeing. The high diffusion coefficient also suggests the possibility of a low-temperature dyeing process.

The Practical Application of Procion Dyes

From the principles outlined in the foregoing section it has been found possible to develop practical methods for applying Procion dyes to cellulose in dyeing, by both continuous and batchwise processes, and in printing. These various methods will be discussed under the above three headings.

I. CONTINUOUS DYEING

In applying these reactive dyes continuously the two stages of dyeing and fixation may be carried out separately or simultaneously. The two-stage process is the easier to understand but more complicated in practice. In a two-stage process the cloth is first padded through a neutral solution of the dye and then through an alkaline solution. It is then subjected to a heat treatment, washed off, and dried. Numerous variations on this theme can be devised. One method is to pad the cloth through a neutral solution of Procion dye, dry, then pad through a solution of caustic soda saturated with common salt, pass through a steamer, wash off in an open soaper, and finally dry. In this process the low affinity of the dye gives very little tailing in the first padding. Tailing is a little more pronounced with the Yellow than with the Red and the Blue, as would be expected from the higher affinity of the Yellow, and is more noticeable on cotton than on viscose rayon. The reason for this is that the dyes in absence of salt have some slight affinity for cotton but none for viscose rayon. In the subsequent drying stage the low affinity of the dyes may permit unlevel migration to take place if the heating is not uniform. Relatively slow drying at a low temperature is desirable, and flue drying is preferable to the use of cans. Addition of electrolyte or of a thickening agent to the padding liquor helps to reduce this tendency to migration. In this connection drying can be avoided by using a "wet on wet" process if a sufficient degree of expression can be obtained in the first pad.

The next stage of this process entails padding the impregnated cloth through alkali. If dilute caustic soda alone is used, the low affinity of the dyes allows bleeding into the padding liquor to occur, and to minimise this the alkali must be loaded with added electrolyte.

Steaming is not particularly critical, but short times only are required, about 15–60 sec. being sufficient. Finally, the fabric must be washed free from loose dye, and here a very efficient washing is required with an adequate supply of water in the early stages. The reason for this is that the fabric is saturated with salt, which increases the affinity of the loose dye, so that it must be removed before the loose dye itself can be removed. Unless the first box of the open soaping range is fed continually with fresh water, it will quickly become saturated with salt and cease to be of any value in

removing loose dye. After washing, the final drying presents no difficulty, as the dye remaining is fully bound and cannot migrate.

An alternative technique is to use the Standfast molten-metal dyeing machine for the heat-treatment stage, the alkali–salt solution being floated on the entrance leg of the machine. All these methods have been employed successfully in practice, and the most serious difficulty encountered has been to overcome migration in the intermediate drying stage.

The dye solutions used in the above methods are neutral and are reasonably stable. If an attempt is made to add alkali to the solutions in order to obtain a one-stage padding process, it is found that the solutions are too unstable to be of practical value. However, sodium bicarbonate can be added without serious instability, and when fabric impregnated with such a solution is heated to 100°C. or higher, reaction with the Procion dye takes place. Consequently a very simple dyeing process consists in padding the cloth through a solution of the Procion dye containing sodium bicarbonate (10–20 g./litre) together with some electrolyte or sodium alginate as a migration inhibitor, then drying, washing off, and drying again. Fixation occurs during the first drying, which may be carried out in a flue drier or on cans. Again great care must be taken to get uniform drying, and if cylinder drying is employed the temperature of the first few cans must be carefully controlled to avoid actual boiling of the liquor on the fabric. This process has also been used successfully in bulk working, especially with viscose rayon fabrics.

The bicarbonate method may also be combined with the crease-resisting process in a sequence such as—

Pad in dye + $NaHCO_3$
↓
Dry
↓
Pad in crease-resisting liquor
↓
Bake
↓
Wash off
↓
Dry

No intermediate washing is required between drying and entry into the crease-resisting liquor, any loose dye being removed in the final washing off. It is noteworthy that basic crease-resisting resins generally promote the retention of loose dye, presumably in the same way as they would an acid dye; but this is not likely to be serious, as any loose dye which is retained will not stain adjacent cellulosic fabrics during domestic washing.

2. BATCHWISE DYEING

In general, a two-stage process is most suitable for batchwise dyeing with Procion dyes. The dyes are first applied to the textile material from neutral solution, salt being added gradually to build up a final concentration of 15–30 g./litre in the dyebath. During this stage the dye is salted on to the fibre smoothly and uniformly in the same way as a direct dye. Because of the much greater rate of diffusion of these dyes, it is possible to carry out this process cold (below 30°C.) in about 30 min. and yet obtain satisfactory penetration with most fabrics. The use of a low temperature is also beneficial in avoiding premature reaction with the fibre. The exhaustion attained at this stage is still low with Procions Red and Blue, but quite high with the Yellow, which illustrates the different affinity of this dye. The levelling properties of all three dyes up to this point are excellent, as would be expected in view of the low exhaustions.

When the neutral dyeing has proceeded for about 30 min. sodium carbonate is added to the dyebath. In the case of Procion Yellow R most of the dye is already on the fibre, and the alkali produces fixation *in situ*. With the Red and the Blue, however, fixation of the adsorbed dye is followed by a further take-up of dye by the fibre. As already noted (p. 240), it is for this reason that the fixation stage also is best carried out cold, so that decomposition in the dyebath is minimised while further diffusion into the fibre is occurring. A further dyeing period of 1 hr. is sufficient, although with the Yellow alone the process can be completed in a much shorter time. The behaviour of the Procion dyes in this process is clearly shown in the rate-of-dyeing curves (Fig. 2–4).

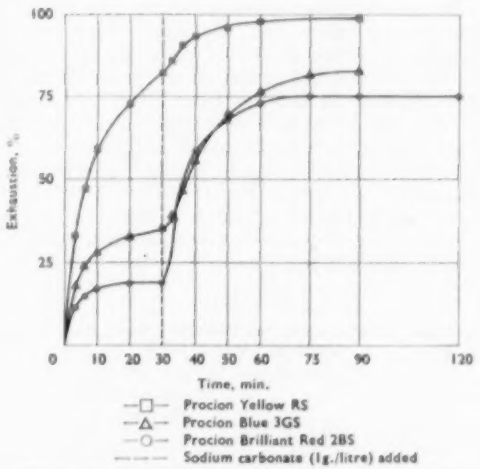


FIG. 2— Rate of Dyeing of Viscose Rayon with Procion Dyes (liquor ratio 30:1, temp. 20°C., 30 g. salt per litre)

In Fig. 2 the rate-of-dyeing curves for the three Procion dyes are displayed. The similarity in behaviour between the Brilliant Red 2B and the Blue 3G and the effect of the higher affinity of the Yellow R are clearly shown. Fig. 3 shows the effect of temperature on the dyeing of Procion Brilliant Red 2B. It will be seen that at the higher temperature a state of equilibrium is reached more quickly in the first stage, but the exhaustion is lower. When alkali is added, reaction is quicker at the higher temperature and is soon complete, but the final amount of dye fixed is much

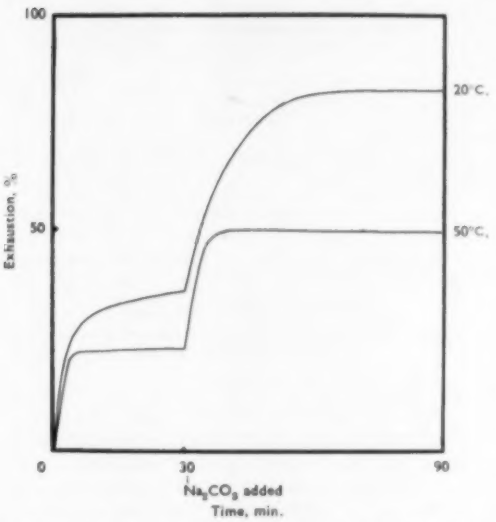
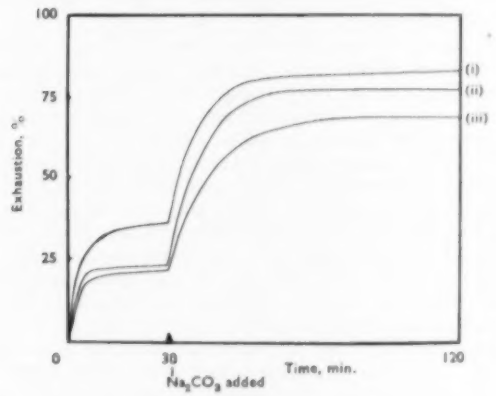


FIG. 3— Effect of Temperature on the Dyeing of Procion Brilliant Red 2B



(i) Viscose rayon
(ii) Mercerised cotton
(iii) Cotton

FIG. 4— Effect of Fibre

less owing to the greater decomposition in the dye-bath. Finally, Fig. 4 shows the relative dyeing properties on cotton, mercerised cotton, and viscose rayon, indicating that the behaviour is in general similar to that of direct dyes, the best fixation being achieved on viscose rayon.

This general process can be used to apply Procion dyes to cellulosic fibres in most types of dyeing machinery with only slight modifications. No difficulty is encountered in yarn dyeing in the beck or in machines of the Hussong type as regards levelness of dyeing. In package-dyeing machines both the salt and the sodium carbonate should be added in portions in order to obtain the maximum levelness, and the rinsing, scouring, and washing-off processes must be particularly thorough. Care must be taken not to leave the finished dyeings in an alkaline condition, as the colour is adversely affected. Winch dyeing may be employed for many types of fabric, the most common fault being rope marking and a lack of penetration or coverage of surface fibres. With fabrics showing these troubles a padding technique may be adopted (see later). The dyes are particularly suited to the dyeing of tufted carpet. Normally, when direct dyes are used, the main difficulty with wide carpeting is side-to-centre shading, largely due to differential temperature effects across the piece. With Procion dyes, dyeing is carried out cold, and such variations are eliminated. In addition, with viscose rayon carpets cold dyeing preserves the crimp and other physical properties of the fibre. No particular difficulties are encountered in jig dyeing.

As already noted, the normal jig and winch methods do not give satisfactory results on certain types of fabric. These are usually materials of tight or dense construction, those woven from tightly twisted yarns, or fabrics, such as linen or spun rayon staple, with surface irregularities. In such cases a pad-and-develop technique gives better results: the goods are first padded through a solution of the Procion dye, and are then passed without drying into a winch or a jig containing a solution of common salt and sodium carbonate to effect fixation. This technique produces dyeings of unsurpassed appearance and solidity with excellent cover of spinning and weaving irregularities, free from listing or ending faults.

3. PRINTING

In printing with Procion dyes one of the first problems encountered was that of finding a suitable thickener. Since most of the common thickening agents are carbohydrates, it is only to be expected that, under any conditions which will lead to fixation of these dyes on the fibre, reaction with the thickener will also occur, and this is in fact observed. The effect is either to give a poor colour yield, since some of the dye is not available to combine with the fibre and is removed in a bound form when the thickener is washed off, or else to insolubilise the thickener in the same way as in the experiment with polyvinyl alcohol (p. 238), so that it cannot be readily removed from the fabric and gives prints with a harsh, stiff handle. The only solution to this problem which has been found so far is the use of alginate thickener. It seems probable that the value of this product lies in the carboxyl groups in the polymer, which ionise and repel the similarly charged dye ions and so prevent reaction. This effect will also promote the transfer of the dye from the thickener into the cellulose.

The printing process is based, again, on the use of sodium bicarbonate. The Procion dye is dissolved in a solution of urea (to improve solubility at the high concentrations required for printing) and added to the alginate thickening. Finally, the sodium bicarbonate is added, if possible just prior to printing. The fabric is then printed in the usual way, steamed, washed off, and dried. The steaming, for 2–15 min., is not critical, and the presence of air does not affect the yield.

The very rapid fixation which is possible with these reactive dyes at high temperatures has led

to the development of a novel printing method which does not involve steaming. This is based on the pad-bicarbonate method of dyeing. The print paste is made up as before, but the sodium bicarbonate may be omitted if desired. After printing, the goods are padded or cover-printed with a thickened solution of sodium carbonate or bicarbonate, and then fixed merely by drying on steam-heated cans. Finally, the goods are washed off and dried.

The various complexities which the practical printer will bring into these simple processes by trying to print Procion dyes alongside other classes of dyes are legion but of no direct interest to the study of reactive systems. The main features of such mixed styles are determined by the pH sensitivity of the Procion dyes and by the chemical stability of the dye molecules from which they are derived. Thus the existing Procion dyes are not stable to reducing agents, which severely limits their use alongside vat dyes, although coloured resists under Procion dyes may be obtained with vat dyes.

In general, it is the fact that Procion dyes will fix only under alkaline conditions which is of major importance in mixed styles. Naturally, they cannot be applied in any style which requires acid steaming, nor can they be very conveniently used alongside dyes which are liable to liberate acid on steaming. Thus, although they can be printed alongside Alcian dyes, special precautions must be taken to avoid contamination of the Procion-printed area with acid or with sodium acetate from the Alcian print paste. Obviously, Procions cannot be used alongside azoic compositions which are to be developed by acid steaming, or alongside solubilised vat dyes developed by the chromate–acid age process. They can, however, be used with neutral-steaming azoic dyes or with solubilised vat dyes applied by the thiocyanate process. By including an excess of sodium bicarbonate in the print paste, Procion dyes may be used as illuminating colours under solubilised vat dyes applied by the thiocyanate process, as the sodium carbonate formed during steaming is sufficient to resist selected solubilised vat dyes.

FASTNESS PROPERTIES OF PROCION DYES

The fastness properties obtained with Procion dyes fall into two groups—those determined by the chemical stability of the dye molecules which form the basis of the Procion dyes, which are independent of the method of linkage, and those which are largely determined by the method of linkage. The existing Procion dyes show a number of deficiencies in the former types of fastness, among which may be mentioned their lack of resistance to hypochlorite; however, such properties are not, of course, characteristic of reactive dyes as a class, but only of those members so far developed. There is every reason to hope that improved products will be found later.

On the other hand, the wet-fastness properties are probably broadly representative of what can be expected from reactive dyes, and are summarised in Table V. The three figures given in each position in the table represent the values for Procions Yellow R, Brilliant Red 2B, and Blue 3G in that order. These fastness figures confirm the behaviour which might be expected of a reactive dye in giving high all-round wet fastness and fastness to solvents. It is notable also that this fastness is maintained at very pale depths of colour, whereas the majority of other dyes show a considerable drop in light and/or wet fastness as the depth is reduced.

TABLE V

Fastness Properties of Procion Dyes[6]

WASHING TESTS

	Cotton			Silk		
	Y	R	B	Y	R	B
	1 × 85°c.			1 × 95°c.		
Effect on dyeing ...	5	5	4–5	5	5	4–5
Staining of cotton ...	4–5	5	5	5	5	5
Staining of wool or silk	5	5	5	5	5	5
	5 × 100°c.			5 × 100°c.		
Effect on dyeing ...	5	3–4	4–5	4–5	4–5	4–5
Staining of cotton ...	4–5	5	5	5	5	4–5
Staining of wool or silk	—	—	—	5	5	4–5

FASTNESS TO DRY CLEANING

No change in colour of dyeing and no staining of adjacent material detectable

CONCLUSIONS

The work which has just been described and the fabrics which have been dyed and printed demonstrate that the chemical reaction of dyes with cellulose is no longer a mere theoretical speculation but a very practical proposition. The Procion dyes place in the hands of the dyer an entirely new method of attaching dyes to cellulose and open a new field of investigation and invention on the application side. Being completely novel, the process has its advantages and disadvantages as compared with conventional methods of dyeing, and its full potentialities have not yet been explored. The use of reactive dyes of the Procion type provides one answer to the age-old problem of making a dye which will be soluble in water and which will have good levelling and penetration properties and so be easy to apply, and yet at the same time give dyeings of the highest wet fastness. The most surprising and gratifying feature is that these effects can be produced with only minor modification of conventional dyeing methods, and that no highly unstable compounds, drastic conditions of reaction, or unorthodox solvents are required. An additional, rather unexpected, feature is the possibility of low-temperature dyeing arising from the rapid diffusion of these dyes into fibres and the ease of reaction. This process is attractive by virtue of the saving of fuel and the general improvement in working conditions in a dyehouse which it can produce.

The Procion dyes themselves must be regarded as the first members only of a potentially very large class of dyes. There can be no doubt that, now the practical utility of the principle has been established, the use of reactive dyes is here to stay, and that the future will see enormous developments in this field, which may well revolutionise certain sections of the dyeing trade.

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[3] Kursanov and Solodkov, *J. Appl. Chem. U.S.S.R.*, **16**, 36 (1943).
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[5] ICI, *Fastness Assessment of Textile Dyestuffs* (2nd edition 1954), Tests No. 3 and 4

DYEHOUSE DEPARTMENT
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DYESTUFFS DIVISION
HEXAGON HOUSE
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(*MS. received 11th March 1957*)

Discussion

MANCHESTER SECTION

Mr. J. GREENWOOD: As shading dyes would need to be applied when the dyebath is alkaline, is it possible to control shading with Procion dyes?

Dr. VICKERSTAFF: With the Red and the Blue, shading dyes may be added directly to the alkaline dyebath without difficulty. In the case of the Yellow, however, its higher affinity might give rise to an uneven strike if this method were used, and it is therefore best to drop the dyebath and add the shading dye to a fresh bath. Since the bath is cold and contains only salt and later sodium carbonate, this is not unpractical.

Mr. K. M. SHAH: What is the relationship between the depth of colour obtained with this class of dyes and that obtained with other classes, and also what is the maximum depth of Procion dyes that can be applied in dyeing and printing?

Dr. VICKERSTAFF: The tinctorial value of Procion dyes is good, and the maximum economic depth of colour is probably about 6%.

Mr. P. MOLYNEUX: What is the cost of Procion dyes in pence per yard of finished fabric as compared with vat or azoic dyes?

Dr. VICKERSTAFF: No complete answer to this question can be given, as the costs differ according to the method of application of the Procion dyes and the individual vat or azoic dyes with which the Procions are to be compared. In dye cost the Procion dyes are generally somewhat cheaper than vat dyes in the yellow, orange, and red ranges, but are more expensive in the blue and green range owing to the relative cheapness of vat dyes of these hues. They are on the average more expensive than direct dyes and considerably more expensive than azoic dyes. They are generally cheaper than solubilised vat dyes. In considering these costs, however, the additional savings on fuel and on chemicals must be borne in mind.

Mr. A. K. TAYLOR: What is the detailed cause of the increase in the adsorption of a Procion dye on addition of alkali to the dyebath?

Dr. VICKERSTAFF: Before addition of alkali the dyeing system is almost in a state of equilibrium, dye molecules diffusing from the dyebath into the fibre and an equal number diffusing out of the fibre into the dyebath. When alkali is added the dye molecules inside the fibre almost instantaneously combine chemically with cellulose, so that the flow of dye molecules out of the fibre ceases. Consequently more dye diffuses into the fibre from the dyebath and is fixed in turn, leading to an increased overall adsorption.

Mr. J. N. MCKEAN: Is it possible to use Procion dyes as a basis for illuminated discharge prints?

Dr. VICKERSTAFF: The existing Procion dyes are sensitive to reducing agents, so that they cannot be used as illuminating colours in a discharge paste. On the other hand, it is possible to use Procion dyes as resists under solubilised vat dyes and Aniline Black, or alternatively, vat dyes can be used as illuminated resists under Procion dyes applied subsequently by padding.

Mr. R. L. HILL: Does the availability of the hydroxyl groups in different cellulose fibres affect the yield and the build-up of Procion dyes, and with Procion dyes are sufficient hydroxyl groups still available after dyeing for further reaction with e.g. resin finishes?

Dr. VICKERSTAFF: The availability of the hydroxyl groups does influence the absorption of Procion dyes, as is shown by the greater fixation obtained on viscose rayon compared with cotton. The hydroxyl groups combined with Procion dyes in a dyeing of normal depth form only a very small proportion of the total number available and do not interfere with any subsequent resin treatment.

Mr. G. S. MARR: Dr. Vickerstaff has emphasised that the properties of Procion dyes indicate low-temperature dyeing as the most desirable procedure, but, on the other hand, he has recommended the Standfast molten-metal high-temperature technique for continuous dyeing.

Dr. VICKERSTAFF: The choice of a low-temperature method depends on whether dyeing is being carried out in a long liquor or not. In a long liquor the exhaustion is low, and in order to get a reasonable yield it is necessary to keep the dyebath cold, so that decomposition with water is reduced to a minimum. This allows further diffusion of dye to take place when alkali is added to the dyebath. On the other hand, in a continuous dyeing process the fabric is padded through the dye liquor, and all the dye is already in the fibre before treatment with alkali and heat. Consequently, the amount of dye which is lost by reaction with water in the fibre is negligible, and a high-temperature dyeing process becomes practicable.

Mr. A. BUTTERWORTH: Has it yet been established in bulk practice that Procion dyes can be satisfactorily applied to cotton materials on the winch, and can Procion dyes be used in a copper vessel in presence of Calgon T?

Dr. VICKERSTAFF: Satisfactory results have been obtained in bulk dyeing with Procion dyes on the winch. As regards the use of metal vessels, the dyes are sensitive to copper and iron, but can be applied satisfactorily in presence of Calgon T or other sequestering agents. On the whole, however, it is best to avoid the use of copper or iron vessels.

Mr. H. W. BEST-GORDON: Does the insolubilisation of cellulose by the dye have any effect on the take-up of urea–formaldehyde resins?

Dr. VICKERSTAFF: The effect is negligible.

Dr. W. SHAW: Does dyeing with Procion dyes, particularly in full colours, have any effect on subsequent finishing to produce e.g. crease-resistance or permanent glaze? Also, do the physical properties of the finished fabric differ from those of fabrics dyed with more conventional dyes?

Dr. VICKERSTAFF: Tests so far have indicated that Procion-dyed fabric does not differ in any significant respect from fabric dyed in other ways as regards subsequent resin finishing. Dyed fibres show a slight loss in extensibility and tensile strength, but this is normally less than 5% and is very similar to that produced by dyeing with vat dyes.

Mrs. M. WHITTAKER: Does the addition of salt to the padding liquor increase the possibility of tailing in continuous dyeing, and if so, is it possible to avoid the addition of salt?

Dr. VICKERSTAFF: The affinities of Procion Brilliant Red 2B and Procion Blue 3G are so low that the presence of salt does not produce any significant tailing. On the other hand, with the Yellow there is a definite increase in the amount of tailing if salt is present, and this may be avoided by using a thickening agent such as sodium alginate as an alternative migration inhibitor.

Mr. H. A. TURNER: If there is more than one reactive group in the molecule of the Procion dyes, is it possible that simultaneous reaction with the fibre and with a molecule of some other dye can occur—for example, could afterdyeing with a Procion dye on a direct-dyed fabric increase the fastness to washing of the direct dye?

Dr. VICKERSTAFF: This may be possible if the direct dye contains a hydroxyl or an amino group, but no direct evidence on this point is available.

HUDDERSFIELD SECTION

Mr. G. ENGLAND: If a union material such as wool–cotton or wool–viscose rayon is treated with Procion dyes, will the dye go on to the cellulosic component first?

Dr. VICKERSTAFF: The answer depends on the condition of the wool. With virgin wool the cellulosic fibre will generally be dyed more heavily, and in such cases the wool can be filled in subsequently with a milling dye to give a union fabric with a very high standard of wet fastness. On the other hand, if the wool is badly damaged or reclaimed wool, then it will tend to dye more heavily than the cellulosic component, and under such conditions it is impossible to fill in the cotton to get a satisfactory overall result.

Mr. H. E. WOOD: Are the Procion dyes pre-metallised, and can development be carried out with gaseous ammonia?

Dr. VICKERSTAFF: The dyes are not pre-metallised. They can be developed by steaming in presence of ammonia.

Mr. R. BUTCHER: Why do the Procion dyes alter the solubility of cellulose in cuprammonium solution?

Dr. VICKERSTAFF: Each Procion dye contains two reactive chlorine atoms, which under cold dyeing conditions both react with the cellulose, so that the dye molecule acts as a cross-linking agent.

Mr. F. DYSON: What is the effect of acid stripping on the colour of Procion dyes?

Dr. VICKERSTAFF: Procion dyes can be stripped to pale grounds in the normal way with reducing agents, and a considerable reduction in depth of colour can be obtained by boiling in dilute acetic acid, which appears to hydrolyse the dye–fibre linkage. By this method the depth is reduced with no effect on the hue.

Mr. ENGLAND: Are the performance of Procion dyes in three-colour combinations and the reproducibility of colour satisfactory, particularly in view of the different properties of the Yellow?

Dr. VICKERSTAFF: The three Procion dyes are perfectly compatible with one another, and because of the low exhaustion in presence of salt and the relative insensitivity to slight variations in salt concentration they are extremely reproducible in colour. This reproducibility extends to three-colour combinations such as greys.

MIDLANDS SECTION

Mr. A. W. CARPENTER: What is the effect of hard water on Procion dyes?

Dr. VICKERSTAFF: These dyes are extremely soluble and are insensitive to hard water up to 10° hardness. In still harder water a loss in colour value may be noted, but this can be avoided by the use of a suitable metal-sequestering agent.

Dr. A. DATYNER: Are reserve effects possible with mixtures of cotton and fibres which do not contain hydroxyl groups?

Dr. VICKERSTAFF: Extremely good reserve effects are possible even on secondary cellulose acetate and also on cellulose triacetate. The fact that no dyeing occurs with secondary acetate is possibly due to the electrical charge developed by this fibre in water, which prevents entry of the Procion dyes.

Mr. L. A. TELESZ: Is the cuprammonium fluidity of cotton restored when a Procion dyeing is stripped?

Dr. VICKERSTAFF: Procion dyeings cannot be stripped with solvents, but only with acid. If dilute acid is used, then dye is not completely removed; while if the strength of the acid is increased, then the cellulose itself is damaged by the acid.

Dr. W. L. LEAD: Now that a group reactive with cellulose has been discovered, it may be possible to use this in other directions, such as with a long fatty chain to give a bound water-proofing finish.

Dr. VICKERSTAFF: Progress in such directions is possible.

A MEMBER: In dealing with very tightly woven constructions which are difficult to penetrate, can the bath be raised to the boil and then allowed to cool before adding alkali?

Dr. VICKERSTAFF: This is undesirable, since Procion dyes are not particularly stable at the boil even in neutral solution. In such a case it is

preferable to pad the dry fabric through a cold solution of the dye and then develop the fabric on the winch or the jig.

Dr. LEAD: The Lecturer has stated that Procion dyes react with amine groups, but there appears to be no reaction with urea in printing.

Dr. VICKERSTAFF: Urea is not sufficiently basic, for no reaction has been observed even with large quantities of urea.

Mr. CARPENTER: What is the behaviour of Procion dyes towards— (*a*) oxycellulose, (*b*) uneven mercerisation, and (*c*) dead cotton?

Dr. VICKERSTAFF: Procion dyes behave in a similar manner to direct dyes, giving paler dyeings on oxycellulose; on unevenly mercerised fabric, spots and marks are developed; while the Procion dyes do not cover dead cotton very well.

Some Observations on the Dyeing of Dicel and Tricel Yarns and Fabrics

H. C. OLPIN and J. WOOD

Meetings of the Scottish Section held at the St. Enoch Hotel on 11th December 1956, Mr. W. G. B. Grant in the chair; and of the West Riding Section held at the Victoria Hotel, Bradford, on 10th January 1957, Mr. C. W. Green in the chair

Full colours are obtainable on Dicel and Tricel with disperse, azoic, and developed dyes. A carrier technique, no longer considered necessary for Dicel, is valuable for Tricel, for which high-temperature dyeing also can be used. Azoic combinations, applicable to both fibres by the concurrent technique, show variable resistance to permanent change of colour on ironing. Some combinations are substantially free from this trouble, but in all cases where it takes place it is associated with change in the degree of aggregation, which suggests redispersion of the dye aggregates in the fibre.

Developed navy blues and blacks are more satisfactory than direct dyeings, and 3-hydroxy-2-naphthoic acid can be used as a developer for both navy blues and blacks on Tricel owing to its greater resistance to change in colour with alkali on that fibre.

Disperse dyes of the C.I. Disperse Blue 27 type are acidic, and their alkali-metal salts are very vulnerable to destruction by light and by oxidising agents, which imposes restrictions on their usefulness on Dicel. On Tricel, however, where salt formation does not take place, these blues are quite safe for all purposes.

1. Introductory Remarks

In the present paper we propose to deal with several topics bearing on the dyeing of Tricel* cellulose triacetate textile material, comparing its behaviour with that of Dicel* secondary cellulose acetate textile material. The properties of cellulose triacetate textile material are given in the literature by Fortess [1] and Stoll [2] in America, and by Boulton [3] and Mellor and Olpin [4] in this country, but it may be profitable to recapitulate its salient features. Generally, it resembles the secondary acetate in behaviour, but it is more hydrophobic, and more difficult to saponify and to delustre. It is dyed with disperse dyes, the majority of which dye at much slower rates and do not achieve such high saturation values. It is also thermoplastic and by suitable heat-treatment can be set in the sense that it is then much less readily softened by heat or water, a property which it shares with the nylons and Terylene. X-Ray diffraction photographs reproduced in the papers referred to above show clearly that this thermosetting of cellulose triacetate is due to increase in the crystallinity of the fibre, as an increase in resolution of the X-ray diagram is observed after such treatment. A consequence of the foregoing behaviour of cellulose triacetate is that effects produced on textiles by hot-embossing, hot-pleating, and similar treatments are very much more resistant to removal by washing than similar effects produced on secondary cellulose acetate textiles.

Tricel is generally acknowledged to be more difficult to dye than Dicel, but less difficult than Terylene and the acrylic fibres. Like Terylene, however, it can be dyed at temperatures above 100°C., this increase in temperature being accompanied by increase in rate of diffusion into the fibre (upon which rate of dyeing depends), and also by increase in the solubility of the dye in the fibre. An explanation of these phenomena has recently been given by Bird [5] in an explanatory paper on the theory of dyeing of cellulose acetate. We have found these theoretical concepts very useful in clarifying certain aspects of the application of azoic dyes to Tricel, which will be dealt with in the appropriate section.

The use of carriers in the application of disperse dyes to cellulose acetate is not new. Before comminution of the disperse dyes had reached its present high standard, the presence of large particles was an obstacle to the realisation of full tinctorial value, and therefore to reproducibility of colour. A considerable volume of work was done by Ellis [6] and his co-workers, who found that dispersions of various organic solvents very appreciably increased the rate of dyeing. Celascour, a proprietary dispersion of xylene in Turkey Red Oil, has been in continuous use on secondary cellulose acetate for thirty years, in the dual rôle of scouring and dyeing assistant. These auxiliary solvents, or carriers, lost importance as better dispersions of the dyes became available. During recent years the use of carriers for the application of the disperse dyes to the polyester fibres has been

**Dicel* and *Tricel* are registered trade marks of British Celanese Ltd. for textiles.*

much publicised, but in acknowledging the vast amount of work carried out in this field, the work of the earlier pioneers on secondary cellulose acetate is also worthy of mention.

In this paper we propose to review briefly the use of disperse dyes on Tricel cellulose triacetate fibres and Dicel secondary cellulose acetate fibres, to discuss methods of obtaining developed navy blues and blacks, and to give an account of some work we have carried out on the properties of the azoic dyes. We also propose to discuss the sensitivity of blues of the C.I. Disperse Blue 27 type to oxidation and to light fading when in the ionised state.

2. Full Colours with Disperse Dyes

2.1. DISPERSE DYES ON DICEL

The disperse dyes are in general use for all kinds of goods made from Dicel yarns, and are capable of giving the widest range of colours. The potentialities of any particular dye for use in obtaining full colours are limited by the all-important factor of solubility of dye in the fibre. The simpler the structure of the dye, the more likely it is to have useful solubility in the fibre, and to have simple light-absorption characteristics. Therefore, we do not find disperse dyes which give truly tertiary colours such as greys, browns, navy blues, and bottle greens. For these colours mixtures of dyes must be resorted to. A great deal of work has been done by dye manufacturers to produce mixtures which will give the requisite colour and fastness, and will also have satisfactory dyeing characteristics such as tone-in-tone build-up, and freedom from listing and ending.

The introduction in the post-war years of a group of disperse dyes of superior fastness to light and gas-fume fading is another step forward, and the American dye industry is to be congratulated on the excellent work it has done in introducing such dyes as—

Eastman Fast Blue GLF	C.I. Disperse Blue 27
Eastman Fast Violet 3R GLF (TE)	—
Eastone Fast Red N GLF (TE)	—
Eastone Fast Brilliant Red 2B GLF (TE)	—
Eastman Fast Yellow GLF	C.I. Disperse Yellow 33
Eastman Fast Yellow 2R GLF (TE)	—
Celanthrene Violet BGF	C.I. Disperse Blue 8
Interchem Acetate Scarlet GSF (IC)	—

It is usual to produce blacks and navy blues on Dicel by applying a disperse diazo component, which is subsequently diazotised and developed with the appropriate developer. Standard navy is obtained with—

S.R.A. Navy Base VI—→Navy Developer I
(C.I. Disperse Blue 11—→C.I. Developer 9)

and standard black with—

S.R.A. Fast Black IV—→3-Hydroxy-2-naphthoic acid
(C.I. Disperse Black 1—→C.I. Developer 8)

We will now examine how closely these general principles can be applied to cellulose triacetate.

2.2. DISPERSE DYES ON TRICEL

One of the advantages claimed for Tricel is its suitability for permanent pleating, and indeed for thermosetting treatments generally, whether for added stability, ease of manipulation, or ease of care of the finished garment. It follows that the dyes chosen for its coloration must withstand these operations satisfactorily: they must not change in colour, nor must they mark off on to adjacent fabric or on to the papers and cardboards normally used in the operation. No degree of finality has yet been reached regarding heat-setting, and the treatments given vary from one establishment to

TABLE I

Disperse Dyes having Superior Fastness to Thermosetting on Tricel

Dye	*Colour Index* No.	Colour Change		Staining on Tricel	
		Dry Heat	Steaming	Dry Heat	Steaming
Amacel Yellow CW	C.I. Disperse Yellow 37	5	5	5	5
Dispersol Fast Yellow A	C.I. Disperse Yellow 1	4–5	5	4–5	4–5
Eastman Fast Yellow 2R GLF (TE)	—	5	5	5	4–5
Dispersol Fast Orange Brown RN	C.I. Disperse Orange 5	4–5	4–5	4	4
S.R.A. Scarlet III	C.I. Disperse Red 7	5	5	5	4–5
Cibacet Brilliant Scarlet RG	C.I. Disperse Red 12	5	5	5	4–5
S.R.A. Red VIII	C.I. Disperse Red 17	4–5	5	4–5	4–5
Dispersol Fast Red R	C.I. Disperse Red 19	4	4	4–5	4–5
Dispersol Fast Crimson B	C.I. Disperse Red 13	5	5	4–5	4–5
S.R.A. Rubine IX	C.I. Disperse Red 5	5	5	5	4–5
Eastone Fast Red GLF	C.I. Disperse Red 35	5	5	4–5	4–5
Eastone Fast Brilliant Red 2B GLF (TE)	—	5	4–5	5	4–5
S.R.A. Fast Violet Blue FSI	C.I. Disperse Blue 19	5	5	4–5	4–5
Artisil Direct Blue GFL	C.I. Disperse Blue 20	5	5	5	5
Serilene Dark Blue G	C.I. Disperse Blue 35	4–5	4–5	4–5	4
Duranol Blue 2G	C.I. Disperse Blue 24	4–5	4–5	4	4
Serisol Fast Blue Green B	C.I. Disperse Blue 7	4–5	4–5	5	4–5
S.R.A. Navy Base VI→Navy Developer I	C.I. Disperse Blue 11 →C.I. Developer 9	4–5	4–5	5	4
S.R.A. Fast Black IV→3-Hydroxy-2-naphthoic acid	C.I. Disperse Black 1 →C.I. Developer 8	4–5	4–5	5	4

another. The overriding consideration, however, is permanence of set, compelling the dyer to suit his selection of dyes to meet these more exacting requirements. Typical test conditions are as follows—

DRY HEAT SET

The sample is sandwiched between two pieces of undyed Tricel, and an 8-lb. iron heated to 200°C. is allowed to stand on it for 30 sec. Change in colour and marking off are noted, using the appropriate grey scale.

STEAM SET

The sample is sandwiched between two pieces of undyed Tricel, and steamed for 10 min. at 10 lb./sq.in. pressure. Change in colour and marking off are noted. This test may very conveniently be carried out in a domestic pressure cooker, if the samples are well wrapped.

These conditions may vary slightly from those usually given in practice, but since the aim is classification and selection, the deviation is excusable. Some of the fastest of the disperse dyes are given in Table I.

It is not claimed that this list is complete, but it is indicative of the type of dye which is likely to give most satisfactory results.

2.3. USE OF CARRIERS

Many of the dyes given in Table I have poor dyeing characteristics on Tricel, and dyers have been searching anxiously for methods of increasing both speed of dyeing and amount of dye absorbed by the fibre. Carriers have given promising results, but they are not popular owing to the various snags and pitfalls inseparable from their use. They are present in the dyebath, and, being mainly organic liquids having poor solubility in water and being very good solvents for disperse dyes, are apt to cause dye spots and other troubles unless handled with great care. They vary greatly in their efficacy, which depends on their ability to swell the fibre sufficiently to allow greater speeds of migration within the fibre. Solubility of dye in the carrier, and of the carrier in the fibre, are both important, the latter especially so when preset fabrics are being processed. Diethyl phthalate has been studied[4] and is of interest because it is representative of a group of carriers which assist the dyeing of preset fabrics, which normally have reduced capacity for dye. Tri-*n*-propyl phosphate, which is in use in America, on the other hand, is a very much milder swelling agent, and appears to be limited in its action to the disoriented portions of the fibre. It is therefore much more valuable on unset than on preset fabrics. It is thus advisable to examine a carrier on both unset and preset fabric, in order to decide whether it is the best for a particular purpose.

2.4. SWELLING TREATMENTS

The chief objection to carriers of the insoluble ester type is their substantivity, which poses the problem of their removal without removal of dye at the same time. To be readily removable from the fibre the swelling agent must be soluble in water, but high concentrations of solvent in the dyebath would then be necessary, and the process would become uneconomical. It is well to remember, therefore, that our objective is to open up the structure of the fibre so that dye can enter more freely and can migrate more rapidly in the fibre. It has been found that fibres so swollen do not contract to their original condition when the swelling agent is removed by washing with water, but remain appreciably hydrated, in which state, according to the degree of initial swelling, they can be as receptive of dye as Dicel. A few examples of such swelling agents are acetic acid, acetone, and ethyl lactate. A very useful swelling agent is diethylene glycol diacetate ("DEGDA"), which is miscible with water in all proportions, has a gentle swelling action on the Tricel fibre, and may be used over a wide range of concentrations and temperatures. Padding cold with 60% DEGDA, and then after a very short time interval washing in cold water, is very effective, and fabric so treated can be dyed in the same manner as Dicel. Treatment in 30% DEGDA at 60°C. for 2 min. gives a similar result.

These pretreatments are also effective for developed navy blues and blacks, which are discussed in the next part of this paper.

3. Developed Navy Blues and Blacks

3.1. BLACKS ON TRICEL BY THE NORMAL SEQUENTIAL METHOD

Our standard black on secondary cellulose acetate has long been S.R.A. Fast Black IV Base (C.I. Disperse Black 1) diazotised and coupled with 3:2-hydroxynaphthoic acid, and naturally it comes under consideration for Tricel.

It has been observed on p. 248 that fastness properties of colorations on Tricel must be of a very high standard in order to give satisfaction in heat-setting and steam-pleating. Blacks are no exception, and combinations which would probably be satisfactory on Dicel are suspect on Tricel. The actual colouring matters we require are in themselves of sufficient fastness, but it is difficult to obtain them free from yellow impurities arising probably from decomposition of the diazotised base. Bases of the *p*-phenylenediamine→dialkylaniline type appear to give this trouble, however carefully they are applied, and bases of the S.R.A. Fast Black IV type are slightly susceptible, though probably acceptable if carefully applied. There are, however, one or two bases the diazo compounds of which are considerably more stable than the diazo compounds of the bases mentioned above, and it is possible to apply them, diazotise, and couple sequentially with 3-hydroxy-2-naphthoic acid as on Dicel. These bases are of the *pp'*-diaminoazobenzene type. They are readily dispersible, and have good affinity for the fibre, but are rather slow-dyeing and do not exhaust well owing to their relatively high solubility in water. A base which is of interest in this connection is that obtained by coupling diazotised *p*-nitroaniline with 3-acetamido-6-methoxyaniline and reducing the nitro group. This base has an exceptionally stable tetrazo compound, which couples cleanly with 3-hydroxy-2-naphthoic acid by the sequential method to give blacks which are of a pleasing blue–black tone and are fast to heat-setting and steam-pressing.

Eastman Black SN (TE) (C.I. Disperse Black 9) is another base which can be diazotised and coupled in this manner, but it is also relatively soluble in water, so exhaustion is not high. These bases are mentioned, however, to show that these problems of dye chemistry are not insuperable, and to suggest that in time the dyer will be provided with bases having the properties he requires. A recipe for applying this type of black is given as Example 1 in the Appendix.

3.2. BLACKS ON SWOLLEN TRICEL

It has been mentioned in § 2.4 that swelling pretreatments, e.g. with aqueous DEGDA, open up the fibre structure, so that Tricel can be dyed with disperse dyes substantially as can Dicel. This applies also to the various black bases, which are in fact disperse dyes, and the benefit of the pretreatment is also carried forward to the developing stage, which proceeds normally. A recipe for applying S.R.A. Fast Black IV developed with 3-hydroxy-2-naphthoic acid is given as Example 2 in the Appendix.

3.3. BLACKS BY THE TWO-BATH CONCURRENT METHOD

This can conveniently be regarded as a variant of the established method of applying azoic dyes to cellulose acetate fibres. For manipulative reasons it has not been found satisfactory to apply both base and 3-hydroxy-2-naphthoic acid together, but it is possible to apply them stepwise, following which diazotisation and coupling proceed simultaneously. This method has been applied successfully in bulk, again using S.R.A. Fast Black IV and 3-hydroxy-2-naphthoic acid. A recipe is given as Example 3 in the Appendix, from which it will be seen that a liberal amount of base is required to compensate for losses during the application of the developer.

3.4. AZOIC BLACK ON TRICEL

It is possible to obtain good blacks on Tricel with the bases discussed above using the simpler arylides of 3-hydroxy-2-naphthoic acid, especially the *o*-toluidide and the *o*-anisidide. Even at the boil, however, the uptake of the arylide is very slow, and although results are satisfactory in the laboratory, the more exacting conditions in the dyehouse demand revision of this simple procedure. In 1955 it was pointed out [4] that an excellent nigger brown can be obtained with the azoic combination 2:5′-dimethoxy-2′:5-dimethyl-4′-aminoazobenzene and Naphtol AS–DB (C.I. Azoic Coupling Component 16), using diethyl phthalate as carrier. The technique can also be applied to blacks with satisfactory results. Diethyl phthalate is not difficult to emulsify with the usual dispersing agents, and it is also available as a 66% proprietary emulsion. Diazo and coupling components are applied together from a neutral bath in the presence of the diethyl phthalate, and diazotisation and coupling proceed simultaneously in the usual diazotising bath. A recipe is given as Example 4 in the Appendix.

3.5. NAVY BLUES WITH S.R.A. NAVY BASE VI (C.I. DISPERSE BLUE 11)

Our standard navy on Dicel at Spondon is obtained by applying S.R.A. Navy Base VI, diazotising, and coupling with S.R.A. Navy Developer I (C.I. Developer 9). This gives a very good navy, which has a fastness to light of 6 or better according to depth, is fast to wet treatments, and is immune to gas-fume fading. It deserves to be far more widely used than it is at present. It is not so easy to apply to Tricel, however. It is true that the diazo component is slower-dyeing on Tricel than on Dicel, but this for once is welcome, since it is absorbed very rapidly by Dicel, and the slower rate on Tricel permits greater ease of control of levelness. Rates of diazotisation and coupling, too, are very much slower, and adjustments must be made. Even so, it is difficult to obtain complete coupling before some of the diazo compound decomposes, and a more rapid developer is needed. S.R.A. Navy Developer III, very similar to S.R.A. Navy Developer I but with a higher coupling potential, gives satisfactory results, but has not, as yet, been brought into general use. The important point is that a satisfactory developed navy blue is possible on Tricel, and will doubtless be made available when the demand is sufficiently great. A suitable procedure is given as Example 5 in the Appendix. It should be mentioned, however, that S.R.A. Navy Base VI can be developed quite satisfactorily with S.R.A. Navy Developer I if the fabric is given the preswelling treatment with aqueous DEGDA discussed in § 2.4.

3.6. NAVY BLUES WITH 3-HYDROXY-2-NAPHTHOIC ACID AS DEVELOPER

We have spent much time examining the possibilities of using 3-hydroxy-2-naphthoic acid in view of the fact that navy blues obtained with this developer, which are invariably sensitive to alkali on Dicel, are quite free from this defect on Tricel. All the black bases give more or less desirable navy blues at about half strength, and they may come under consideration when maximum light fastness is not essential. Unfortunately, we have been unable to find any of these couplings which have a light fastness of more than 3–4 in light navies, and 4 in normal depth. Work on this problem is still in progress.

3.7. SUMMARY

Blacks can be obtained on Tricel with a limited number of bases, which can be applied, diazotised, and developed sequentially as on Dicel. Preswelling in e.g. 60% DEGDA enables Tricel to be dyed substantially as Dicel with disperse dyes generally, and also with developed navy blues and blacks. The so-called two-bath concurrent method is also useful, both black base and 3-hydroxy-2-naphthoic acid being applied separately, following which diazotising and developing proceed simultaneously. Azoic blacks can also be produced with the same bases, but using suitable arylides of 3-hydroxy-2-naphthoic acid. Carrier technique greatly assists uptake of the arylide.

Navy blues can be obtained on both Dicel and Tricel with S.R.A. Navy Base VI and S.R.A. Navy Developer III by the usual sequential method, but S.R.A. Navy Developer I is unsuitable for Tricel unless a swelling pretreatment has been given. Many bases give good navy blues on Tricel with 3-hydroxy-2-naphthoic acid. On this fibre they are not sensitive to alkali, but fastness to light leaves much to be desired.

4. Azoic Dyes on Dicel and Tricel

4.1. VALUE OF AZOIC DYES ON THE CELLULOSE ACETATE FIBRES

The azoic dyes have been used on Dicel for many years, mainly to obtain colours fast to wet processing, but also to obtain bright reds and clarets not readily obtainable in the requisite fullness by other means. Browns have been obtained by combining Naphtol AS–LB (C.I. Azoic Coupling Component 15) with various diazo components, and Naphtol AS–DB (C.I. Azoic Coupling Component 16) has also been used. The possibilities of azoic combinations on Tricel have been indicated previously [4].

Although it was considered possible that the introduction of spun-dyed Dicel would reduce interest in the yarn-dyed material, it has been found in practice that the two are complementary, and considerable weights are still being dyed with azoic and vat dyes, and are marketed as our Class A range. In this field hank dyeing is giving place to package dyeing, with corresponding improvement in yarn quality. It is significant that a number of manufacturers now market azoic diazo components and arylides of 3-hydroxy-2-naphthoic acid and similar hydroxy-carboxylic acids ready mixed for application by the concurrent process. Among such azoic compositions now available are—

Acetazols (Fran)
Brentacets (ICI)
Ofna-cets (FH)
Silatose Azoics (JWL)

The concurrent process involves application of both components together from aqueous dyebaths, treating them as disperse dyes. The colour is developed by immersion in a diazotising bath, in which diazotisation and coupling proceed simultaneously, though this is not always the case, a notable exception being Fast Blue BB Base (C.I. Azoic Diazo Component 20), which has a low coupling potential, and is fully coupled only in a subsequent alkaline treatment.

Dyeing at temperatures above 100°c., e.g. in the temperature range 115–125°c., is impracticable for Dicel, but is undoubtedly of value for Tricel. Its economic value for the latter in fabric form is still under investigation, but for package dyeing of yarn it has many advantages. Very useful and informative accounts of the application of azoic dyes to secondary cellulose acetate and Terylene have been given by Walls [7] and by Fern and Hadfield [8]. So many factors have to be taken into consideration when applying azoic dyes to hydrophobic fibres that it is not surprising that these workers have left some important points still to be clarified. We now propose to describe some work we have carried out on the aggregation of azoic dyes on both Dicel and Tricel, and a detailed investigation into the behaviour of Fast Red B base (2-methoxy-4-nitroaniline) on Tricel showing how uptake of this base varies with temperature and concentration.

4.2. DYEING OF TRICEL YARN AT HIGH TEMPERATURES

With increase in temperature of the dyebath the following benefits accrue—

(*a*) Imbibition of water by the fibre is increased, though not to such an extent as to cause collapse of the fibre, the package (cheese or cone) remaining rigid and permeable throughout.

(*b*) Decrease in viscosity and surface tension of the dye liquor with rise in temperature ensures a more rapid flow for a given pressure head, as will be appreciated by reference to Table II.

TABLE II

Viscosity of Water

Temp. (°c.)	Viscosities relative to—	
	Water at 0°c.	Water at 80°c.
0	1·000	—
40	0·367	—
60	0·262	—
80	0·199	1·000
100	0·158	0·795
124	0·124	0·627

(*c*) Migration of dye within the fibre, which is a most important factor regulating the rate of uptake of disperse dyes by Tricel, is greatly accelerated [9].

(*d*) Solubility of dye in both media is increased.

The last-named factor is one which does not always operate in favour of the fibre. The final state in the dyeing operation is one of dynamic equilibrium of dye between dyebath and fibre, but with increase in temperature solubilities increase in both media. If, however, solubility in the dyebath increases at a greater rate than solubility in the fibre, uptake of dye by the fibre could be adversely affected, especially at high liquor ratios. This depends on the initial solubility in water and the partition coefficient between dyebath and fibre. With these considerations in mind we took Fast Red B Base (C.I. Azoic Diazo Component 5—2-methoxy-4-nitroaniline) as our model and obtained the relationships given in the series of graphs in Fig. 1–4.

Fig. 1 shows the straight-line relationship between the reciprocal of the absolute temperature and the logarithms of the solubilities of the base in dyebath and fibre respectively. Fig. 2 shows how partition of base between dyebath and fibre moves in favour of the dyebath at higher temperatures. Fig. 3 shows the straight-line relationship between the logarithm of the partition coefficient K and the reciprocal of the absolute temperature, as required by the equation—

$$\ln K = -\frac{\Delta G}{RT}$$

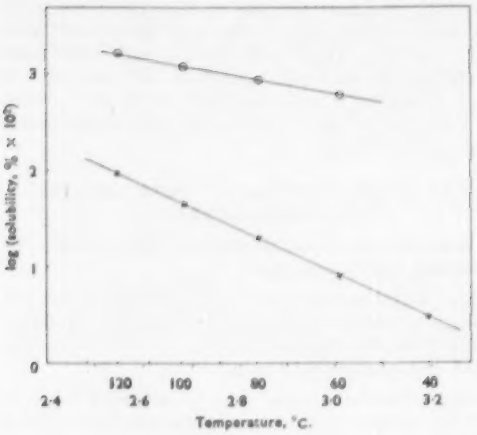


FIG. 1—Solubilities of 2-Methoxy-4-nitroaniline at Various Temperatures

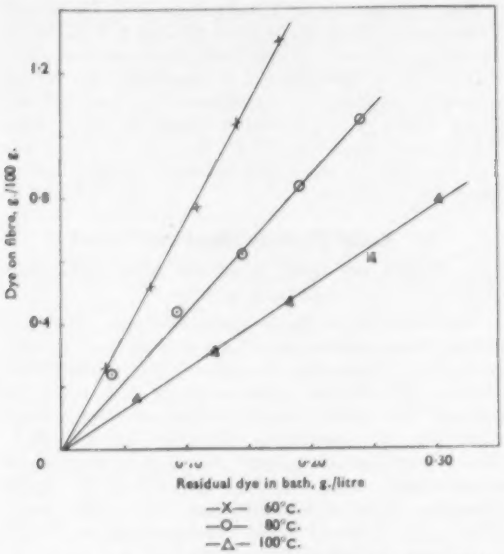


FIG. 2—Distribution of 2-Methoxy-4-nitroaniline between Tricel Fibre and Dyebath at Different Temperatures

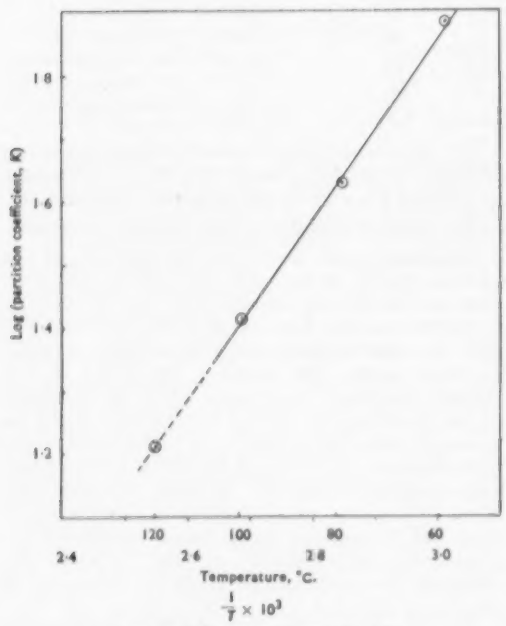


FIG. 3—2-Methoxy-4-nitroaniline on Tricel

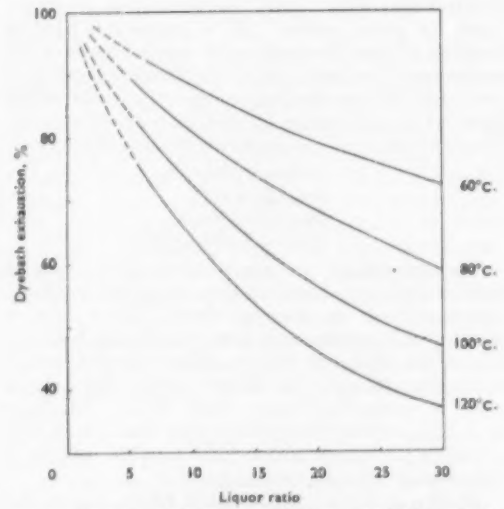


FIG. 4—Variation of Dyebath Exhaustion of 2-Methoxy-4-nitroaniline with Liquor Ratio at Specified Temperatures

and in Fig. 4 these data are collected into a working chart from which percentage exhaustions of the diazo component at various temperatures and liquor ratios can be ascertained by inspection.

Table III shows the solubilities of Fast Red B Base in the dyebath and in the fibre.

It is noteworthy that, although this base is very soluble in the fibre, solubility in the dyebath is also a very important factor. With this particular base the complication of saturation of the fibre does not arise, but with less soluble bases and disperse dyes charts such as Fig. 4 are subject to this limitation.

This study was found to be time-consuming, but from these data and our experience with other water-soluble bases we felt justified in concluding that a working solution to the problem of concurrent application of diazo and coupling components to Tricel would be to dye at the higher temperature first, in order to ensure uptake of

TABLE III

Saturation Values of 2-Methoxy-4-nitroaniline on Tricel at Various Temperatures

Temp. (°c.)	Solubility (g./litre)	Partition Coefficient (K)	Saturation Value (%) (calc.)
40	0·28	—	—
60	0·76	76·2	5·8
80	1·96	42·5	8·3
100	4·46*	25·8	11·5
120	9·23*	17·0*	15·7

*Estimated

sufficient coupling component, subsequently allowing the diazo component to be absorbed in a cooling bath.

4.3. HEAT SENSITIVITY OF AZOIC DYES

4.3.1. Heat Sensitivity of Azoic Dyes on Dicel

This phenomenon was first noticed on a piece of Dicel fabric dyed to a cherry red with the combination—

Fast Red B Base—→Naphtol AS–LT
(C.I. Azoic Diazo Component 5—→C.I. Azoic Coupling Component 24)

which had been aggregated, but not to its final colour. A hot iron placed on this fabric caused a permanent change of colour towards scarlet, not to be confused with the temporary change of many dyes, which is reversible on conditioning.

This permanent change could, however, be reversed by resoaping, this fact alone pointing to the probable cause, viz. deaggregation of dye at temperatures at which the fibre is thermoplastic and is acting as a solvent for the dye. It seemed probable that the phenomenon was not confined to this particular combination, so a range of combinations were dyed in medium to full depths on Dicel fabric in the normal manner, and portions treated in soap (2 g./litre) for 1 hr. at 60°c. and 80°c. respectively. The patterns were dried without ironing, and tested by applying a stationary iron at 190°c. for 10 sec. Results are given in Table IV.

From Table IV it will be seen that heat sensitivity varies from very severe to nil, and is generally less severe with dyeings soaped at 80°c. than with those soaped at 60°c. Couplings with Naphtol AS–LB (C.I. Azoic Coupling Component 15) seem to be in a class by themselves, since they change colour on soaping, and yet are not sensitive to heat.

4.3.2. Effect of Various Aftertreatments

A fuller investigation of the effect of the aftertreatment given to a full depth of red on Dicel obtained with the combination Fast Red B Base→Naphtol AS–LT gave the results listed in Table V.

TABLE V

Effect of Aftertreatments on Fast Red B Base→Naphtol AS–LT on Dicel

(C.I. Azoic Diazo Component 5—→C.I. Azoic Coupling Component 24)

Agent	Concn. (g./litre)	Temp. (°c.)	Colour	Heat Sensitivity
Adinol T	1	60	Cherry red	Very severe
Adinol T	1	70	Slightly bluer	Pronounced
Adinol T	1	80	Distinctly bluer	Moderate
Adinol T	1	90	Much bluer	Slight
Soap	5	70	Slightly bluer	Pronounced
Soap	5	80	Distinctly bluer	Moderate
Soap + Celascour	2 3	80	Distinctly bluer	Moderate
Soap	2	98	Much bluer	Nil

It is clear from this table that the temperature of the aftertreatment is the important factor, and that heat sensitivity can be practically eliminated by soaping at 90–98°c. Unfortunately, this cannot always be achieved in practice, and in any case it is accompanied by marked colour change and delustring of the fabric.

4.3.3. Heat Sensitivity of Different Azoic Combinations

A large number of dyeings were examined, and a few general tendencies were noted, although sensitivity varied from member to member—

(*a*) All the combinations tested using aminoazobenzene as diazo component were much less sensitive than those from nitro-amino bases, such as Fast Red B or Fast Red 2G Base.

TABLE IV

Heat Sensitivity of Azoic Combinations on Dicel

Constitution		C.I. Azoic Diazo Component No.	C.I. Azoic Coupling Component No.	Colour at		Heat Sensitivity at	
Base	Naphtol			60°c.	80°c.	60°c.	80°c.
Fast Red KB	AS–G	32	5	Lemon	Little change	Slight	Slight
Fast Red B	AS–G	5	5	Yellow	Slightly redder	Moderate	Slight
Aminoazotoluene	AS–G	4	5	Golden yellow	Little change	Moderate	Moderate
Fast Scarlet GG	AS–BG	3	19	Orange–red	Duller	Pronounced	Pronounced
Fast Red RC	AS–OL	10	20	Scarlet	Bluer	Moderate	Slight
Fast Red B	AS–LT	5	24	Cherry	Bluer	Very marked	Pronounced
Aminoazotoluene	AS–LT	4	24	Wine	Slightly bluer	Moderate	Moderate
Fast Red GG	AS–LB	37	15	Fawn	Slightly redder	Nil	Nil
Fast Scarlet GG	AS–LB	3	15	Yellow–brown	Little change	Nil	Nil
Fast Red RL	AS–LB	34	15	Brown	Very slightly yellower	Nil	Nil
Fast Red B	AS–LB	5	15	Red–brown	Yellower	Nil	Nil

(*b*) Naphtol AS–LB couplings were not sensitive with any of the bases tested.

(*c*) All the bright red azoic combinations tested showed moderate to severe sensitivity.

Among those tested were several red and wine compositions from the dyemakers' ranges, which are listed in Table VI.

TABLE VI

Heat Sensitivity of Commercial Products on Dicel

Product	*Colour Index* No.	Colour	Sensitivity
Silatose Azoic Red GL (JWL)	—	Pink	Very slight
Silatose Azoic Bordeaux GL (JWL)	—	Bluish pink	Slight
Brentacet Red B (ICI)	—	Scarlet	Pronounced
Brentacet Bordeaux B (ICI)	—	Wine	Pronounced
Acetazol Red B	C.I. Disperse Red 33	Bluish red	Moderate–pronounced
Acetazol Bordeaux JB	C.I. Disperse Red 36	Bordeaux	Moderate
Ofna-cet Red BL	C.I. Azoic Red 62	Red	Very pronounced
Ofna-cet Red RL	C.I. Azoic Red 61	Scarlet	Very pronounced
Ofna-cet Bordeaux BR	C.I. Azoic Red 64	Claret	Very pronounced

4.3.4. Influence of Depth of Dyeing

The combination—

Fast Red B Base → Naphtol AS–LT

was dyed in depths ranging from full red to pale pink and given the standard aftertreatment of 2 g. soap per litre for 1 hr. at 80°c. The results in Table VII suggest that in very pale dyeings aggregation does not take place on soaping.

TABLE VII

Influence of Depth of Dyeing on Heat Sensitivity

Azoic Components (%)	Colour	Sensitivity
3·0	Full red	Very pronounced
2·0	Full red	Very pronounced
1·0	Medium red	Pronounced
0·5	Deep rose	Moderate
0·2	Pink	Slight
0·05	Pale pink	Nil

4.3.5. Summary of Observations on Dicel

A number of sensitive and non-sensitive dyeings on Dicel were examined by preparing longitudinal sections. Non-sensitive dyeings, e.g. couplings with Naphtol AS–LB, showed no sign of particles either when finished cold or when finished at 95–98°c., but sensitive dyeings, whilst showing definite particles at the higher temperature, were clear when finished cold. The available evidence enabled us to reach the following conclusions—

The phenomenon of change of colour of azoic combinations on ironing is a common one, and is connected with the degree of aggregation of the pigment. If the pigment is not aggregated at all, as in very pale dyeings, no deaggregation can occur, and ironing has no effect. If it is aggregated to its final colour (usually very near to that obtained on cellulose fibres), the aggregates, probably minute crystals, are sufficiently resistant to redispersion to withstand the ironing treatment, and ironing, again, has no effect. If, however, aftertreatment is stopped at some intermediate stage, redispersion can take place on ironing, with consequent permanent change in colour. Unfortunately, it is difficult to control the aggregation of many combinations so that both stability to heat and freedom from rubbing are achieved.

Aminoazobenzene gives azoic dyeings which are much less sensitive than those from the simpler substituted anilines. This is in accord with the above hypothesis, the dye molecules probably being much less mobile. Azoic dyes from Naphtol AS–LB, however, appear to be free from the trouble, even at the intermediate stages of colour change during soaping.

4.3.6. Heat Sensitivity of Azoic Dyes on Tricel

The heat sensitivity of azoic dyes on Tricel follows much the same pattern as on Dicel. There are notable differences in behaviour, but they all arise from the differences in rates of diffusion of dyes generally in the two fibres. Table VIII shows the effect of aftertreating a full dyeing of Fast Red B Base→Naphtol AS–LT in soap (1 g./litre) for various times and temperatures.

TABLE VIII

Effect of Various Aftertreatments on Fast Red B Base→Naphtol AS–LT on Tricel

Treatment	Colour	Sensitivity
Not soaped	Scarlet	Slight
1 hr. at 95–98°c.	Red	Pronounced
1 hr. at 120°c.	Crimson	Very slight, rubs
2 hr. at 120°c.	Crimson	Very slight, rubs
1 hr. at 130°c.	Crimson	Very slight, rubs
2 hr. at 130°c.	Crimson	Very slight, rubs

This table shows that even the unsoaped pattern is slightly sensitive, suggesting the possibility of the coupling component itself being present and coupled partly as the dimer. Again, sensitivity is greatest in the partly aggregated form, an interesting point being rapid increase in rate of aggregation at temperatures above 100°c.

The sensitivity of a number of combinations after soaping for 1 hr. at 95–98°c. is given in Table IX.

5. Vulnerability of Blues of the C.I. Disperse Blue 27 Type

5.1. CHEMICAL STRUCTURE

Some recent cases of fading on washable Dicel garments dyed with Eastman Fast Blue GLF have led to further investigation of the suitability of this dye for Dicel fabrics destined for sports shirts, pyjamas, etc., which are frequently laundered. Though the information received is meagre, there have undoubtedly been genuine cases of colour

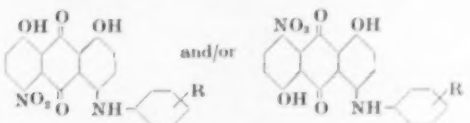
TABLE IX

Heat Sensitivity of Azoic Combinations on Tricel

Constitution		C.I.	C.I.	Heat Sensitivity	
Base	Naphtol	Azoic Diazo Component No.	Azoic Coupling Component No.	Unsoaped	Soaped
Fast Red B	AS–BG	5	19	Very slight	Pronounced
Fast Red B	AS–D	5	18	Very slight	Pronounced
Fast Red B	AS–OL	5	20	Slight	Pronounced
Fast Red B	AS–RL	5	11	Slight	Pronounced
Fast Red B	AS–BO	5	4	Nil	Very slight
Fast Red RL	AS–LT	34	24	Pronounced	Pronounced
Fast Red RC	AS–LT	10	24	Slight	Pronounced
Fast Red KB	AS–LT	32	24	Pronounced	Pronounced
Fast Scarlet RC	AS–LT	13	24	Pronounced	Pronounced
Fast Garnet GBC	AS–LT	4	24	Pronounced	Pronounced
Aminoazobenzene	AS–LT	(1*)	24	Slight	Slight

* C.I. Solvent Yellow 1

destruction during washing (or during steeping before washing), and of accelerated fading of the laundered garments. These blues have the general chemical structure—



according to whether 1:8-dihydroxy-4:5-dinitro- or 1:5-dihydroxy-4:8-dinitro-anthraquinone or a mixture of the two is used. R is a hydroxyalkyl solubilising group. The hydroxy and nitro groups *para* to each other naturally increase the acidity of the molecule, which has been shown by potentiometric titration to give a distinctly sigmoid curve ($pK \simeq 10{\cdot}5$) closely resembling that given by 1-hydroxy-4-nitroanthraquinone. These curves are shown in Fig. 5.

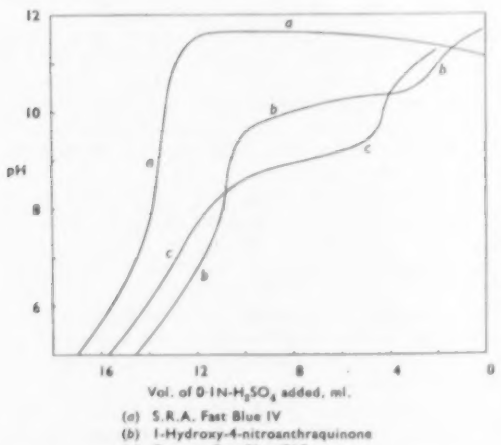
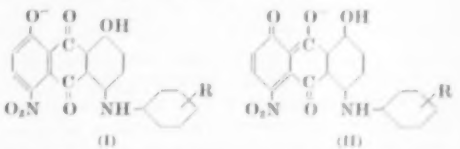


FIG. 5—Potentiometric Titration of Hydroxyanthraquinone Dyes

The procedure was as follows: the dye was freed from dispersing agent and 0·001 mole dissolved in alcohol (100 ml.) and 0·1 N-NaOH (15 ml.) and diluted with water (250 ml.). The solution was then titrated with 0·1 N. sulphuric acid.

The ionised dye can exist in several forms, two of which are given below—



(I) (II)

Form II has lost its essential benzenoid resonance structure, thereby probably becoming more vulnerable to attack by destructive agents.

5.2. LIGHT FASTNESS

The major interest in blues of the "Blue GLF" type is their very high fastness to light and gas fading. The makers' instructions regarding their application ensure that they will be present in the fibre in the normal, or non-ionised, state. Dyeing from the recommended neutral bath is also very much a matter of economics, the dye being soluble in alkaline dyebaths, and equilibrium being displaced very much in favour of the dyebath.

When dyeings of C.I. Disperse Blue 27 are padded with sodium carbonate and exposed to light, the fastness is reduced very considerably. In pursuance of this observation approx. 1% on the weight of the fabric of a number of alkalis was applied to dyeings of this dye on Dicel, dried at room temperature, and exposed to a carbon-arc fading lamp (Fade-Ometer) with the following results—

Padded with	Fading after 25 hr.
Blank (untreated)	Unaffected
Sodium carbonate	Very severe
Sodium bicarbonate	Very severe
Tetrasodium pyrophosphate	Moderately severe
Borax	Moderately severe
Soap	Severe

Another series of tests was carried out as above, but the patterns were thoroughly washed-off in warm water after being treated with alkali. Even under these conditions there was fading after 25 hr. exposure, showing that the sodium salt formed is only slowly hydrolysed by water.

These results, and other trials in which garments were washed in soap, rinsed lightly, and hung up to dry in a manner the housewife is imagined to use, give convincing proof of the vulnerability to light of the alkali-metal salts of blues of this type.

It must be emphasised, however, that "Blue GLF" on Tricel is quite free from this defect, so that it can be used more freely on this fibre than on Dicel.

5.3. DESTRUCTION BY PROPRIETARY DETERGENTS

The instructions on the containers of domestic synthetic detergents are often vague, the general impression being conveyed that no harm could possibly come from their use. One manufacturer advises as follows—

> Soak clothes for 5–10 minutes, then work thoroughly up and down. To clean extra dirty spots rub on dry. . .

Apart from the active detergent and neutral diluents, these proprietary washing powders are liable to contain alkali, usually soda ash, oxygenated compounds, e.g. sodium percarbonate, and fluorescent brightening agents. The last-named are harmless enough, but alkaline oxidising agents were considered worthy of investigation, particularly as the housewife will sooner or later apply the same remedy to coloured garments as is advised for whites. In our experience these proprietary detergents are quite satisfactory for the laundering of cellulose acetate goods, provided that they are used sensibly, and in particular that they are dissolved before use. Unwise use of many of these proprietary detergents, however, and particularly the practice, not unknown among housewives, of sprinkling the detergent on the wetted-out garments, can cause local destruction of the colour. For example, a paste consisting of 0·5 g. of sodium percarbonate, 2 g. of soap, and 10 ml. of water spotted on to medium depths of a number of dyes on Dicel, and allowed to dry in air, resulted in the destruction of a number of disperse dyes, whilst others were quite unaffected. Eastman Fast Blue GLF is among those destroyed in this manner, as also are a number of other anthraquinone dyes containing hydroxy groups, among these being—

S.R.A. Fast Red VII	1-Amino-4-hydroxy-anthraquinone
Eastman Fast Violet 3R GLF (TE)	—
Interchem Acetate Blue GSF (IC)	—
Serilene Dark Blue G (YDC)	—
Celanthrene Violet BGF	C.I. Disperse Blue 8

This possible source of trouble is mentioned because it has given rise to one or two complaints, but we would emphasise that, generally speaking, when used correctly, modern detergents are acceptable for laundering both Dicel and Tricel goods.

This destruction does not occur on Tricel tested in a similar manner, the effect again probably being due to inability of the alkali to penetrate the fibre.

Appendix

1. BLACK ON BRIGHT TRICEL FABRIC BY NORMAL SEQUENTIAL PROCESS (§ 3.1)

Woven fabric dyed on an enclosed jigger in 10:1 bath

Eastman Black SN powder→3-Hydroxy-2-naphthoic acid

(C.I. Disperse Black 9→C.I. Developer 8)

The dyebath is prepared with 10% Eastman Black SN powder calculated on the weight of fabric and 1 g. Lissapol C per litre. Dyeing is commenced at 45–50°c., and the temperature slowly raised to 95°c. and maintained thereat for a further 3–4 hr.

After washing off, the goods are diazotised by treating for 1 hr. at 25°c. in a 10:1 bath containing, per litre—

3 g. ... Sodium nitrite
9 ml. ... Hydrochloric acid (32°Tw.).

The goods are then washed off in a bath containing 0·5 ml. hydrochloric acid per litre.

Development is carried out in a 10:1 bath having the following composition per litre—

3·3 g. ... 3-Hydroxy-2-naphthoic acid
1·1 g. ... Caustic soda
1·0 ml. ... Dispersol VL (ICI)
0·8 ml. ... Formic acid (85%).

The 3-hydroxy-2-naphthoic acid is dissolved in the caustic soda and Dispersol VL, and added to the dyebath, and the pH value is adjusted to 4·0–4·5 with the diluted formic acid. The fabric is entered cold and raised to 75°c. during 30 min., development being continued for a further hour.

After a cold wash-off, the fabric is finally cleared in a bath containing 1 g. Lissapol C per litre for 35–40 min. at 75°c.

2. BLACK ON SWOLLEN TRICEL BY THE NORMAL SEQUENTIAL PROCESS (§ 3.2)

Woven fabric dyed on an enclosed jigger in 10:1 bath

The scoured and dried fabric is padded at 25°c. through a double nip with a liquor containing 60% DEGDA and 2% Celacol MM 10. It is then given two cold-water washes.

The dyebath is set with 10% S.R.A. Fast Black IV 300 powder (C.I. Disperse Black 1) and 1 g. Lissapol C per litre. Dyeing is commenced at 45–50°c., the temperature raised to 80–85°c., and dyeing continued at that temperature for 3 hr. The goods are then washed off thoroughly, and it is advisable at this stage to check for rubbing.

Diazotisation and development are carried out as in Example 1.

3. BLACK ON TRICEL BY TWO-BATH CONCURRENT METHOD (§ 3.3)

Woven fabric dyed on an enclosed jigger in 10:1 bath

11% S.R.A. Fast Black IV 300 powder (C.I. Disperse Black 1) is applied at 95°c. from a dyebath prepared as in the preceding example, and the

fabric is given a cold-water wash, and then treated in a fresh bath containing, per litre—

3 g. ... 3-Hydroxy-2-naphthoic acid
1 ml. ... Dispersol VL

at pH 3·5. The temperature is raised from cold to 75°C. during 30 min., and maintained at this value for a further hour. The goods are rinsed in 0·5 ml. hydrochloric acid per litre, and then diazotised and developed as follows—

The bath is made up with, per litre—

3 g. ... Sodium nitrite
9 ml. ... Hydrochloric acid (32°Tw.)

and the goods are treated at 25°C. for 90 min. They are then washed off, and given a treatment in soda ash (1 g./litre) for 30 min. at 20°C. A final clearing treatment is given in Lissapol C (1 g./litre) at 75°C., any tendency to "crocking" being corrected by addition of an appropriate amount of hydrosulphite and an alkali. The goods are finally washed off in water.

4. AZOIC BLACK ON TRICEL (§ 3.4)

For preset locknit fabric on an enclosed winch in a 30:1 bath

The dyebath is prepared with 12% Brentacet Black B powder, calculated on the weight of fabric, which is pasted with sufficient Lissapol C to give a concentration of 0·5 g./litre in the dyebath, and diluted by adding boiling water. To the dyebath is then added diethyl phthalate (5 g./litre) suitably emulsified with a further 0·5 g. Lissapol C per litre.

Dyeing is commenced at 45–50°C., and the temperature slowly raised to 80–85°C., and maintained thereat for a further 2 hr. After washing off, the goods are diazotised and developed in a bath containing, per litre—

1 g. ... Sodium nitrite
5 ml. ... Formic acid (85%).

The fabric is run for 10 min. cold, then the temperature is raised to 60°C., and the fabric run for 30 min. at 60°C. The goods are then washed off thoroughly, and finally cleared in a bath containing 3 g. Lissapol NC + 10 g. sodium chloride per litre for 30 min. at 75°C.

The goods are finally washed-off in water.

5. NAVY BLUE ON SWOLLEN TRICEL BY THE SEQUENTIAL METHOD (§ 3.5)

Woven fabric dyed on an enclosed jigger in a 10:1 bath

The scoured and dried fabric is padded at 25°C. through a double nip with a liquor containing 60% DEGDA and 2% Celacol MM 10. It is then given two cold-water washes.

The dyebath is set with 3·0% S.R.A. Navy Base VI 300 powder (C.I. Disperse Blue II) and 1 g. Lissapol C per litre. Dyeing is commenced at 45–50°C., the temperature raised to 85°C., and dyeing continued at that temperature for 2 hr.

After washing off, the goods are diazotised by treating for 1 hr. at 25°C. in a 10:1 bath containing, per litre—

3 g. ... Sodium nitrite
9 ml. ... Hydrochloric acid (32°Tw.).

The goods are then washed off, and development is carried out in a bath having the following composition per litre—

2·0 g. ... S.R.A. Navy Developer I (hydrochloride) (C.I. Developer 9)
1·0 ml. ... Dispersol VL
0·5 g. ... Sodium acetate
0·2 ml. ... Glacial acetic acid.

The fabric is entered at 30°C. and raised to 75°C. during 30 min., and development continued for a further hour.

After a cold wash-off the fabric is finally cleared in a bath containing 1 g. soap per litre for 1 hr. at 90°C.

* * *

The authors are pleased to acknowledge the work contributed by their colleagues in the Dye Research Department. They also thank the Directors of British Celanese Ltd. for permission to publish this paper.

A number of processes described in this paper are the subjects of patents pending.

DYE RESEARCH DEPARTMENT
BRITISH CELANESE LTD.
SPONDON
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(*MS. received 18th January 1957*)

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Discussion

(WEST RIDING SECTION)

Mr. A. MURRAY: Have the lecturers found a disperse blue possessing good light and gas-fume fastness on Dicel which is not sensitive to alkali or alkaline detergents?

Mr. OLPIN: We appreciate the difficulties confronting the dye manufacturers in devising blue disperse dyes which are both immune to gas fading and fast to light. We do not wish to condemn blues of the GLF type, but rather to point out a definite weakness for correction in future introductions.

Mr. MURRAY: The lecturers have mentioned that pretreatment with DEGDA increases the uptake of dye of both unset and preset Tricel. Would the lecturers recommend a DEGDA treatment and presetting before dyeing, or is it better to dye the unset fibre?

Mr. WOOD: There is no object in presetting if this can be avoided, since the DEGDA would first

be required to undo the effects of the presetting treatment.

Mr. C. L. BIRD: Is the solubility of disperse dyes inherently lower in Tricel than in Dicel?

Mr. OLPIN: According to Majury [10], who worked with film, Tricel is as good a solvent for disperse dyes as is Dicel. Commercial fibres, however, are probably more highly crystalline than film, and we are examining the possibility that both degree of crystallinity and extent of swelling of the fibre modify the volume fraction of the fibre available for dyeing.

Dr. F. MANCHESTER: Do swelling agents increase the rate of aggregation of azoic dyeings on the secondary acetate and the triacetate?

Mr. WOOD: Yes, the rate of aggregation will be increased, because the dye molecules will be more mobile in the swollen fibre.

Mr. E. STEAD: What effect does diethyl phthalate have on the pleating characteristics of cellulose triacetate?

Mr. OLPIN: If the diethyl phthalate is completely removed from the fibre, its effect on pleating characteristics will be negligible. Owing to its insolubility in water, however, this is difficult to achieve, and any carrier left in the fibre will be reflected in slight lowering of the pleating temperature, depending on the amount present.

Mr. J. H. LEACH: Have swelling agents any effect on differential dye affinity associated with unlevel heat-treatment in presetting? Also, do they tend to produce any alteration or diminution in the colour change on heat-setting after dyeing?

Mr. OLPIN: We have differentiated between swelling agents of the diethyl phthalate type, which apparently swell the heat-set as well as the unset fibres, and those like tripropyl phosphate, which swell only unset fibres. We should expect the former to level out any unevenness due to unlevel heat-treatments, but not the latter. Swelling agents speed up the dyeing operation, so that the system moves nearer to final equilibrium in economic dyeing times in their presence than in their absence. A major cause of colour change during the heat-setting of Tricel dyed with disperse dyes is intensification of colour due to solubilisation of surface-precipitated dye by Tricel in the thermoplastic state. This fault should be eliminated when swelling agents are used.

Dr. C. B. STEVENS: Are the fastness characteristics of triacetate dyeings produced by the preswelling technique using DEGDA in any way inferior to those of dyeings produced without using this solvent?

Mr. WOOD: No, the use of DEGDA results in better dyeings, which are at least as fast as, and often faster to wet processing than, those obtainable on untreated fabric.

Dr. STEVENS: We found that triacetate yarn impregnated with a 50:50 mixture of benzyl alcohol and trichloroethylene, squeezed, and entered into a plain aqueous dyebath containing either Irgalan Grey BL (C.I. Acid Black 58), Irgalan Yellow GL (C.I. Acid Yellow 114), or Irgacet Scarlet RL (Gy) was rapidly dyed at 70°C. In absence of solvent the dye was deposited only on the surface and completely removed by a light scour. Would you expect these metal-complex dyes to behave in this way?

Mr. WOOD: Tricel is analogous to Dicel in that it is capable of absorbing a very wide range of dyes, including the 2:1 metal-complex type, when in the swollen state. This principle underlies all solvent dyeing techniques, and can also be exploited in solvent printing. The dyes mentioned have no substantivity for normal Tricel, but can be applied by swelling techniques.

COMMUNICATION

The Flameproofing of Nylon

D. O. DOUGLAS

Thiourea–formaldehyde resins can be used to give a non-inflammable finish both on nylon net and on nylon dyed with chrome dyes. The mechanism of the flameproofing of nylon is discussed. The effects of flameproofing agents and of chrome dyes on the melting point, the viscosity, and the ignition temperature of nylon are examined. In the case of nylon dyed with chrome dyes it is suggested that effective flameproofing agents counter the fall in ignition temperature by lowering the melting point. In the case of resin-treated nylon it is probable that the lowering of the viscosity in the presence of flameproofing agents assists the dropping away of the melt.

INTRODUCTION

Fabrics made from nylon yarn are not normally inflammable, owing to the fact that the melting point of nylon is lower than the temperature at which it ignites. As a result, when nylon is heated by a flame it melts and recedes from the flame. If the molten nylon is prevented from receding, it burns when it has reached the temperature at which it ignites. An example of this is the behaviour of nylon braid: when a thick nylon braid is heated in a flame, the outside reaches the ignition temperature before the inside has reached the melting point, the inside then acts as a wick for the molten exterior, and the braid burns.

Another well known instance of the modification of the behaviour of nylon is the inflammability of nylon dyed with chrome dyes and afterchromed. The dye itself plays no part, since undyed chromed nylon also is inflammable.

Nylon has proved to be particularly suitable for the manufacture of dress nets for evening wear in place of continuous-filament viscose rayon. For

this purpose nets must be finished stiff, and a durable stiff finish can be produced on nylon by the use of thermosetting resins. Melamine resins were preferred for this purpose because of their better washing fastness[1]. Unfortunately, nylon net finished in this way is highly inflammable.

Thiourea–formaldehyde was originally proposed as a means of flameproofing nylon dyed with chrome dyes[2] and has been used to obtain a non-inflammable stiff finish on nylon[3]. Difficulties in the manufacture of thiourea–formaldehyde resin syrups prevented the general acceptance of the method.

The main object of this work was to find a simple method of producing a flameproof finish on nylon nets, and the most obvious way of achieving this appeared to be to use thiourea as an addition to a commercially available resin precondensate. The work which was done on the production of flameproof finishes was largely concerned with the selection of a suitable resin precondensate, and with a comparison of the tensile and tear strengths, washing fastness, crease resistance, etc. of nylon nets treated by possible alternative procedures with those of nets treated by the established method. The precondensate selected as a result of these tests was a methylated hydroxymethylurea (urea : formaldehyde ratio 1 : 1·9). It was found that thiourea did not give a flameproof finish when added to methylated hydroxymethylmelamine.

The work which is reported here was undertaken in the hope that a clearer understanding of the mechanism by which an addition of thiourea to certain resins produces flameproof finishes would lead to other flameproofing agents or to improvements in methods of applying flameproof finishes containing thiourea.

MECHANISM OF FLAMEPROOFING

Hasselstrom *et al.*[4] examined a number of substances as flameproofing agents for nylon. They concluded that "most conventional flameproofing materials used for other textiles tend to increase the inflammability of nylon, except for certain sulphur or chlorine containing compounds. It is postulated that the latter prevent flame propagation of molten nylon by the fact that they produce acidic sulphur compounds and hydrogen halides on pyrolysis. These, in turn, decompose the ammonium carbonate produced in heated nylon, with the liberation of carbon dioxide. The amount of carbon dioxide may not be sufficient in itself to prevent flame formation. However it may be assumed that the use of flameproofing materials capable of producing acidic sulphur compounds or hydrogen halides may increase the action of the carbon dioxide by preventing the formation of intermediary heat-stable carbonates or acid amides."

In the recent paper by Fishman[3] on the use of thiourea as a flameproofing agent for nylon the suggestion is made that thiourea–formaldehyde has a melting point similar to that of nylon and that this permits resin and nylon to drop off together.

Neither of these explanations seems to be satisfactory. Fishman's explanation does not account for the fact that nylon dyed with chrome dyes is flameproofed by thiourea, nor does it account for the effectiveness of many other substances. The difficulty with the former explanation is that it does not account for the failure of thiourea to flameproof nylon when added to melamine resins.

The common factor in the circumstances which cause nylon to burn was believed to be a restraint on the dropping away of the molten nylon. This appears to be true in the case of resin-treated nylon, but it proved to be a false assumption in the case of nylon dyed with chrome dyes. It seemed that the most probable mechanism of flameproofing is one which involves a lowering of the melting point of nylon with a consequent fall in viscosity of the nylon melt. Hasselstrom[4] reports screening tests on substances potentially suitable for flameproofing nylon fabric and finds that some substances which flameproof nylon raise its melting point, but in no case is the melting point lowered. In view of the importance of this question, the effect of flameproofing agents on the melting point was first examined.

Experimental

INFLUENCE OF FLAMEPROOFING AGENTS ON THE MELTING POINT OF NYLON

Weighed samples of nylon net were padded in 10% solutions of the flameproofing agents, passed through a hand-wringer, dried slowly on a laboratory pin frame to minimise migration, and weighed. The flameproofing agents tested are listed in Table I together with the amounts applied.

Small cuttings of the impregnated nylon together with a blank were placed in short lengths of glass tube, both ends of which were then sealed. The sealed tubes were placed in an oven at 190°C., and the temperature was raised. When the contents of a tube fused it was removed, and the temperature recorded. The results of three sets of tests are given in Table I.

TABLE I

Melting Points of Nylon to which Flameproofing Agent has been added

Flameproofing Agent	Uptake (%)	Melting Points (°C.) Set 1	Set 2	Set 3
Blank	—	245–250	248–253	245–250
Ammonium dihydrogen phosphate	15·8	245–250	248–253	245–250
Ammonium chloride	5·3	230–235	233–238	235–240
Ammonium bromide	10·4	225–230	233–238	235–240
Ammonium sulphate	10·5	235–240	248–253	245–250
Ammonium sulphamate	11·3	230–235	248–253	245–250
Ammonium nitrate	11·3	214–223	228–233	220–225
Ammonium thiocyanate	7·8	200–210	233–238	240–245
Thiourea	10·4	200–210	220–225	220–225

It was understood that the method was not likely to afford highly consistent results, and indeed there is a certain variation in the melting points of the blanks. The variation in the results for nylon treated with flameproofing agents is, however, much greater than would have been expected from the results on the blanks. It was known that the conditions of drying had varied between sets, and this was thought to be a possible reason for the differences. Two sets were prepared: in one case the tubes containing the nylon were stored in a vacuum desiccator over the week-end and afterwards sealed, and in the other a drop of water was placed in the tube before sealing. This procedure can only be said to ensure that of the two sets one was wetter than the other. The melting points of the wet and dry samples were compared (Table II).

TABLE II

Comparison of Melting Points (°C.) of Wet and Dry Samples

Flameproofing Agent	Dry	Wet
Blank	250–255	240–245
$NH_4H_2PO_4$ ...	250–255	240–245
NH_4Cl	235–240	195–205
NH_4Br	235–240	195–205
$(NH_4)_2SO_4$ ...	245–250	195–205
$NH_2{\cdot}SO_2NH_4$ ...	245–250	195–205
NH_4NO_3... ...	230–235	205–215
NH_4CNS ...	240–245	195–205
$CS(NH_2)_2$ ...	235–240	195–205

The depression of the melting point of nylon by flameproofing agents is therefore increased in the presence of water, and the variations in the results of the tests on the dry samples could well have been due to variations in drying. However, if the results of melting-point determinations on dry material are tabulated in the order of the efficiency of the flameproofing agents in lowering the melting point (Table III), it will be seen that the order is reasonably consistent.

TABLE III

Order of Decreasing Efficiency of Flameproofing Agents in lowering the Melting Point of Nylon

(Thiourea most efficient in Sets 1–3)

Set 1	Set 2	Set 3	Set 4
$CS(NH_2)_2$	$CS(NH_2)_2$	$CS(NH_2)_2$	NH_4NO_3
NH_4CNS	NH_4NO_3	NH_4NO_3	$CS(NH_2)_2$
NH_4NO_3	NH_4CNS	NH_4Cl	NH_4Cl
NH_4Br	NH_4Br	NH_4Br	NH_4Br
NH_4Cl	NH_4Cl	NH_4CNS	NH_4CNS
$NH_2{\cdot}SO_2NH_4$	$NH_2{\cdot}SO_2NH_4$	$NH_2{\cdot}SO_2NH_4$	$NH_2{\cdot}SO_2NH_4$
$(NH_4)_2SO_4$	$(NH_4)_2SO_4$	$(NH_4)_2SO_4$	$(NH_4)_2SO_4$
$NH_4H_2PO_4$	$NH_4H_2PO_4$	$NH_4H_2PO_4$	$NH_4H_2PO_4$

The flameproofing agents tested are, with the exception of thiourea, ammonium salts of strong acids. The action of heat on thiourea results in partial isomerisation to ammonium thiocyanate. At equilibrium, the mixture contains 75% ammonium thiocyanate. Findlay [5] published a graph to illustrate the results obtained by measuring the freezing points of mixtures of ammonium thiocyanate and thiourea and determining the concentrations in the cool mixtures. The melting point of thiourea fused at 170°C. given by Prätorius-Seidler [6] (149°C.) corresponds on Findlay's graph to conversion of 30% of thiourea to ammonium thiocyanate. It can be shown by heating nylon net treated with methylated hydroxymethylurea to which thiourea has been added that combination of thiourea with resin does not prevent its conversion to ammonium thiocyanate.

It may be significant that the ammonium salts of volatile acids are more effective than ammonium salts of non-volatile acids. A related observation is that nylon will withstand carbonising with sulphuric acid but not with hydrochloric acid [7]. As a check that the melting point of nylon is lowered by the action of hydrochloric acid, nylon was subjected at 190°C. to the action of a stream of hydrogen chloride gas which had been dried by passing through concentrated sulphuric acid. The product was fused at this temperature after 20 min. exposure. The results obtained are at variance with those reported by Hasselstrom *et al.* [4]

EFFECT OF FLAMEPROOFING AGENTS ON THE INFLAMMABILITY OF NYLON DYED WITH CHROME DYES

An attempt was made to find whether the efficiency of flameproofing agents in lowering the melting point of nylon is related to their efficiency in flameproofing nylon. These tests were made on woven nylon fabric which had been dyed with a chrome dye and afterchromed, since it had been found by preliminary trials that differences were more readily observed on fabric than on net.

Weighed pieces of this fabric were padded in solutions of the flameproofing agents, dried slowly on a pin frame to minimise migration, and weighed.

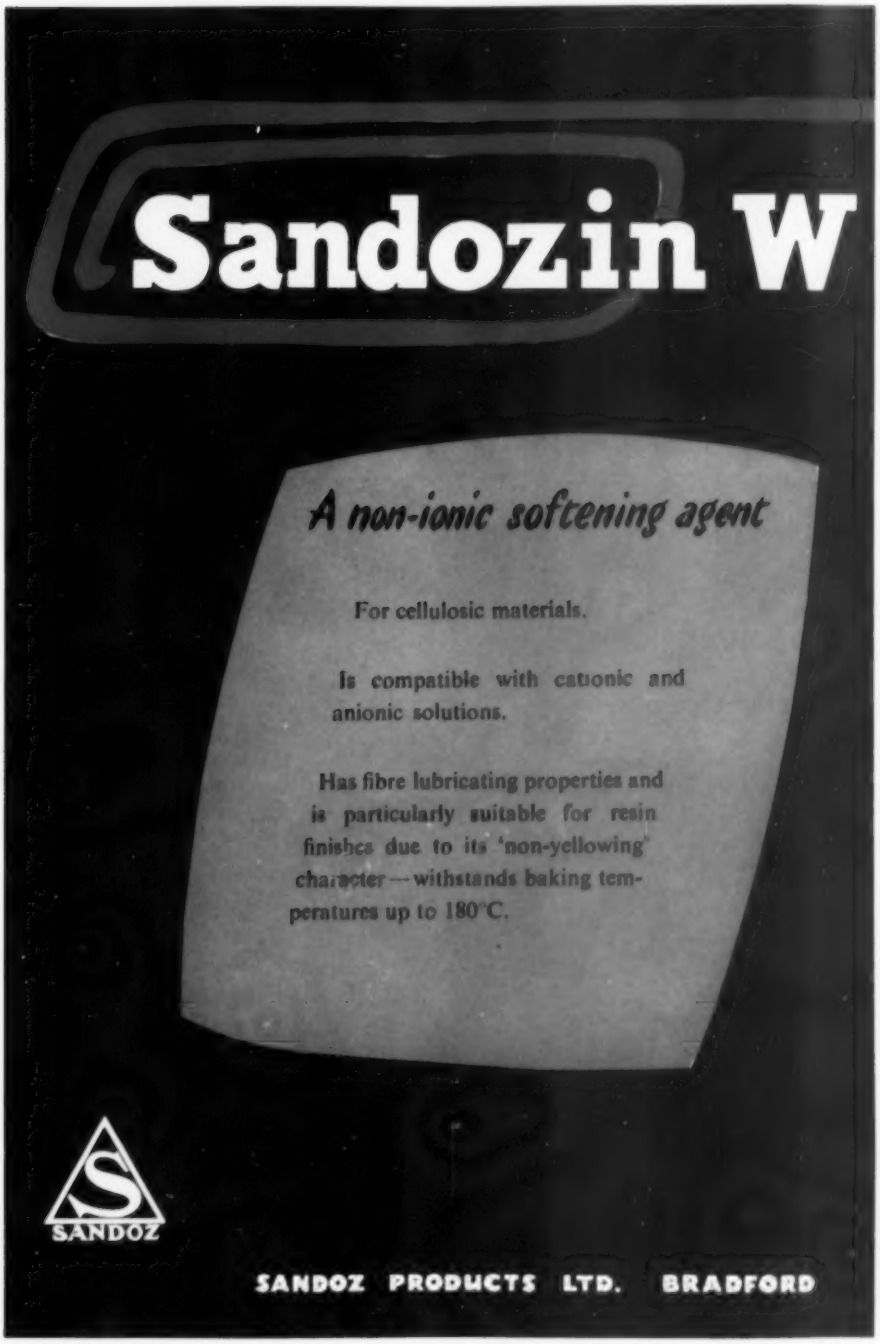
Tests for inflammability were made by hanging a sample vertically and applying a wax taper, as required by the legal standard of non-inflammability [8]. In this way the minimum amount of the flameproofing agent which would flameproof the nylon fabric was determined (Table IV).

TABLE IV

Minimum Effective Amounts of Flameproofing Agents

Flameproofing Agent	Uptake (%)	
Thiourea	3·05	
Ammonium thiocyanate ...	5·91	
Ammonium bromide	9·14	
Ammonium sulphate	11·33	Insufficient to flameproof
Ammonium dihydrogen phosphate	17·7	Insufficient to flameproof

These results indicate that the efficiency of the salts as flameproofing agents is related to their efficiency in lowering the melting point.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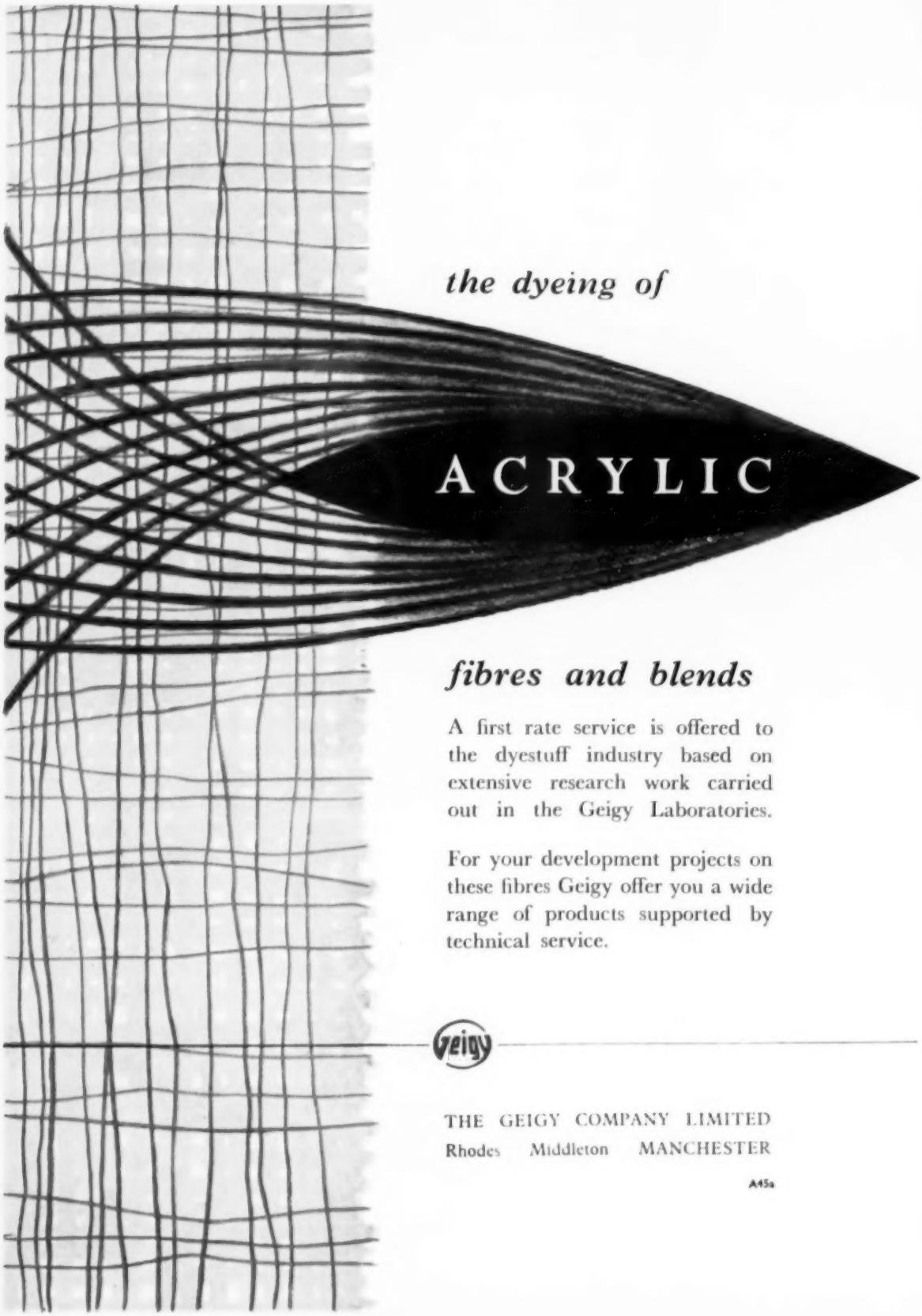

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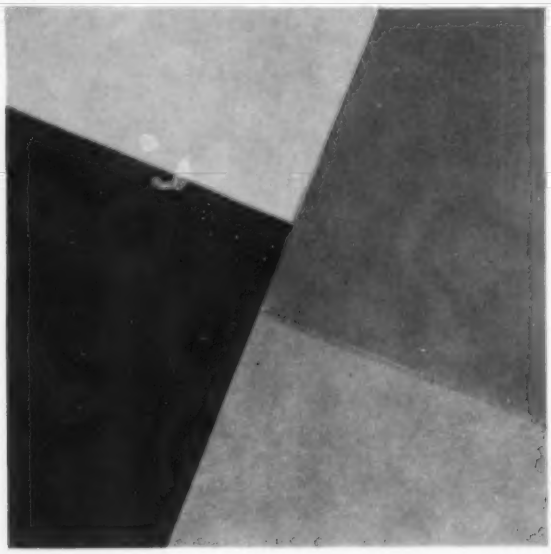

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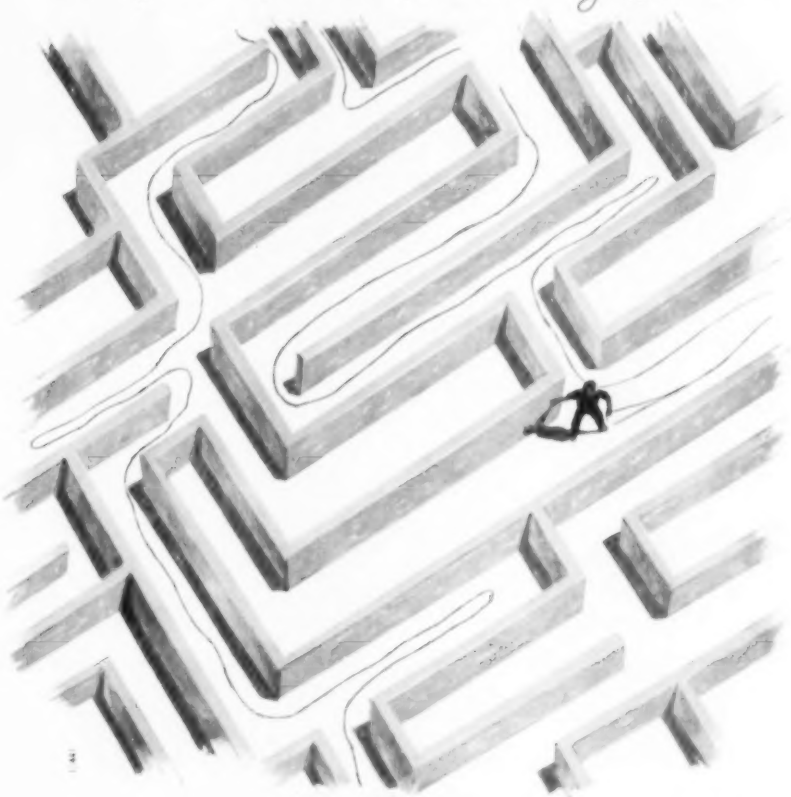

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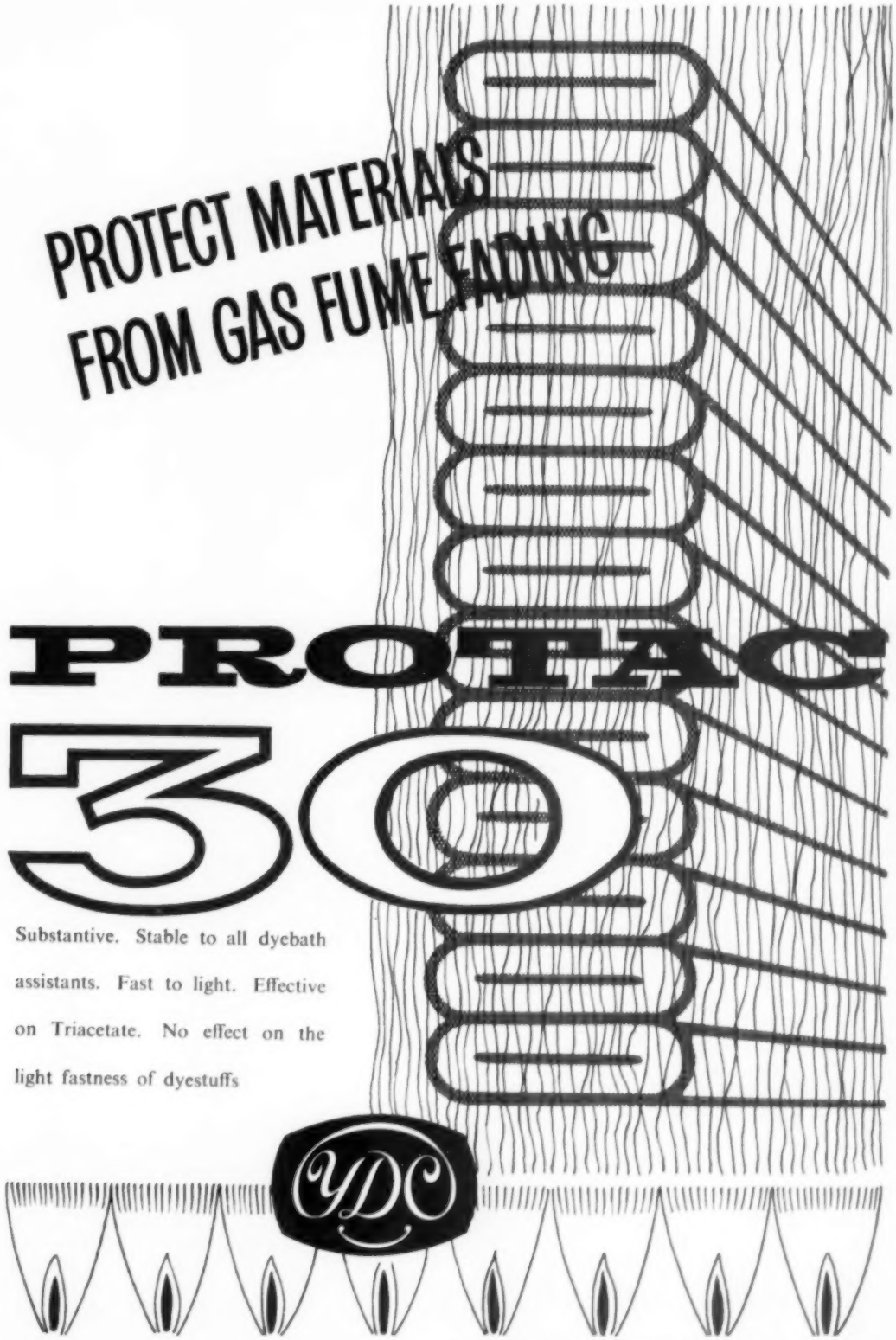
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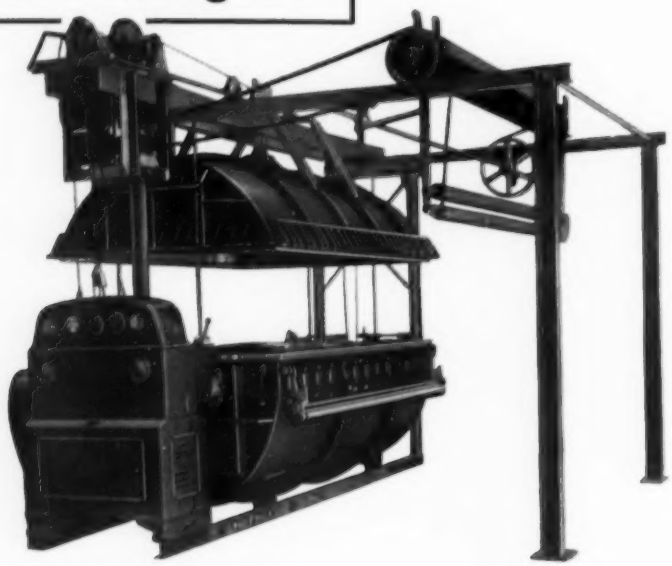

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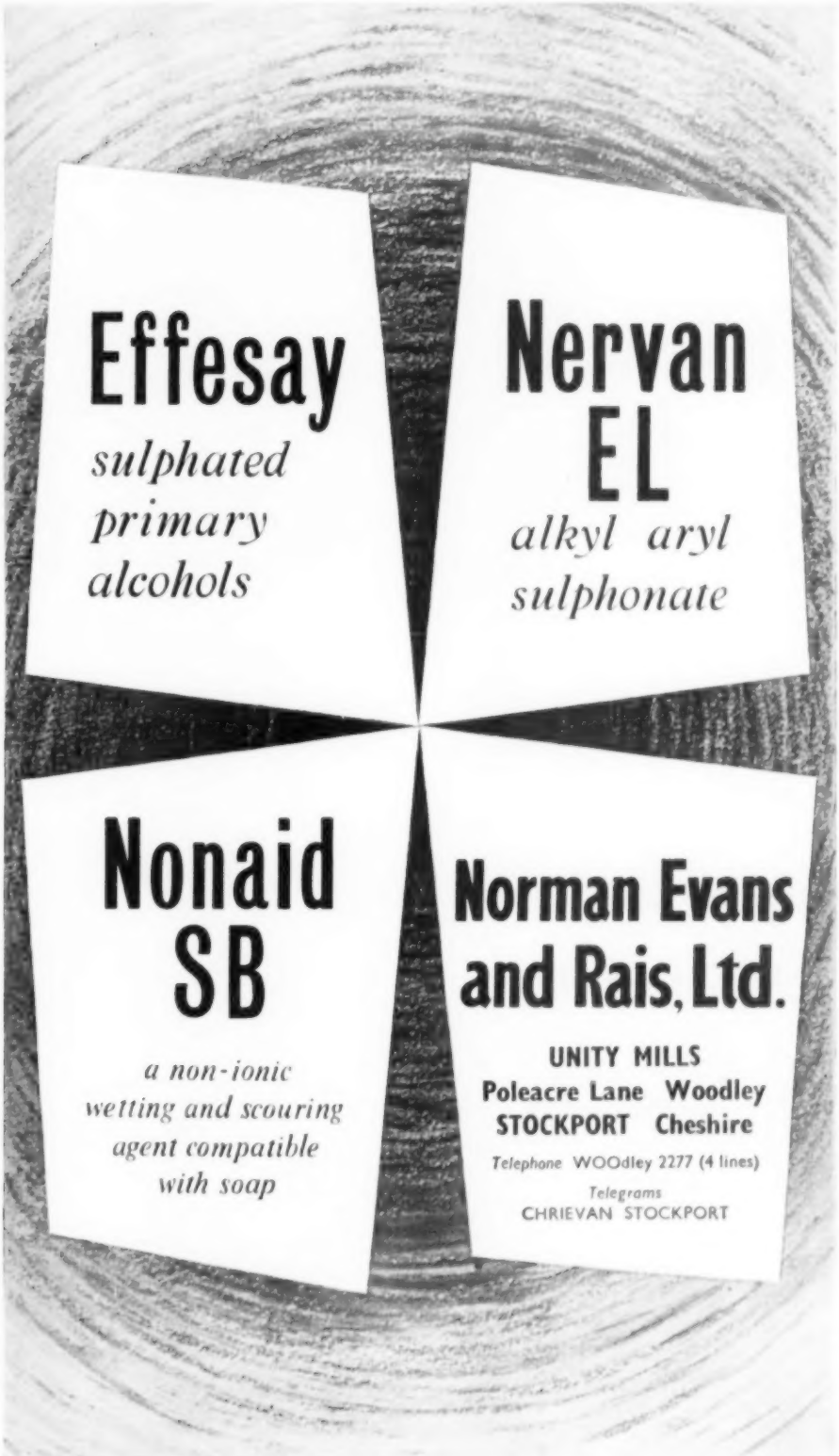
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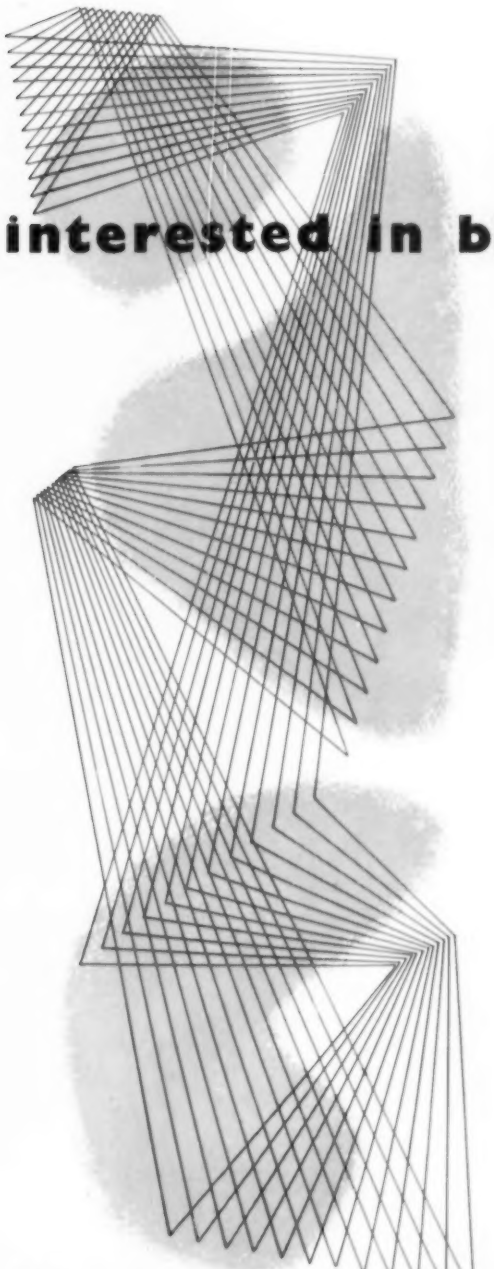

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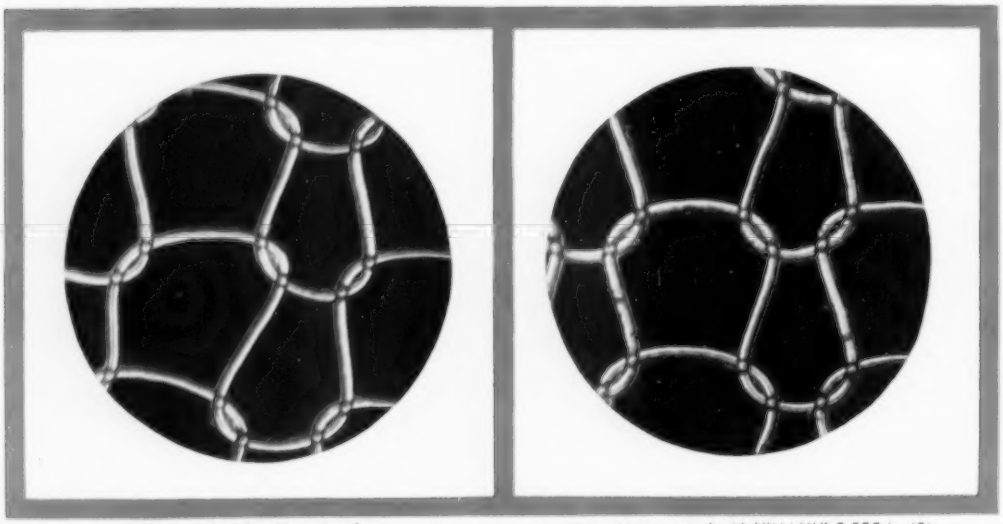

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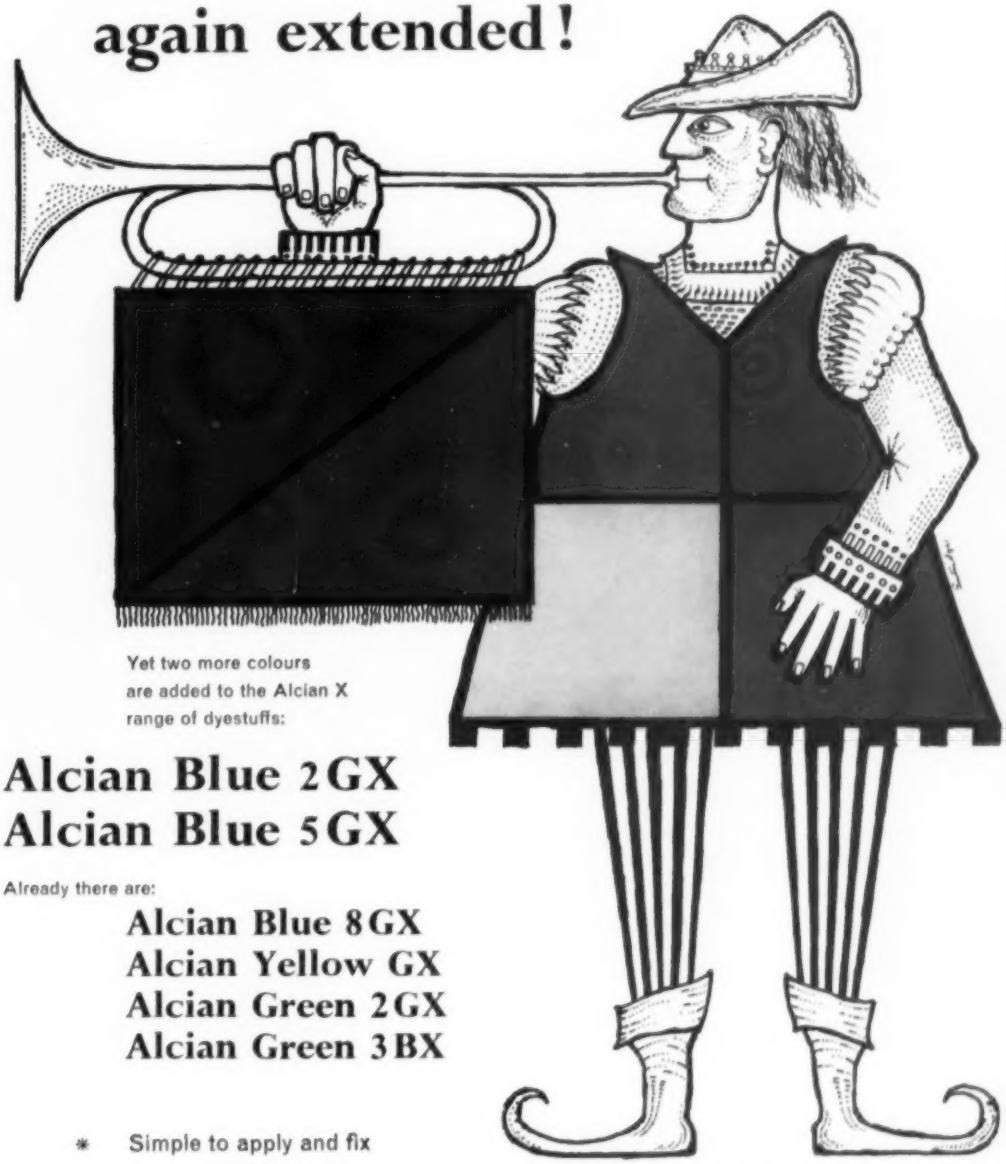

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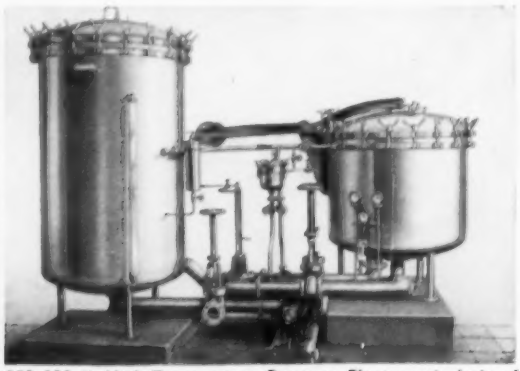

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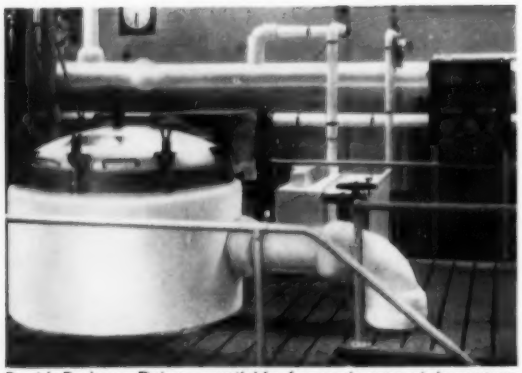

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EFFECT OF FLAMEPROOFING AGENTS ON THE MELTING POINT OF RESIN-TREATED NYLON

In order to find whether the lowering of the melting point of nylon by thiourea is still observed when nylon is treated with resin to which thiourea has been added, samples were finished with methylated hydroxymethylmelamine, methylated hydroxymethylurea, and methylated hydroxymethylurea with addition of thiourea. Melting points were determined as before (Table V). The lowering of the melting point observed with thiourea is observed also when thiourea is included in the resin finish.

TABLE V

Effect of Resin Treatments on Melting Point

Resin Treatment	Melting Point (°C.)
Methylated hydroxymethylmelamine ...	250–255
Methylated hydroxymethylurea	245–250
Methylated hydroxymethylurea with addition of thiourea	235–240

EFFECT OF FLAMEPROOFING AGENTS ON MELTING POINT OF RESIN SOLIDS

Since thiourea is a flameproofing agent only in the presence of urea resins, there is a possibility that thiourea has a specific action on these resins.

In order to observe the effect of flameproofing agents on resin solids, additions of the salts were made to the resin syrups, which were then dried. The dried resins were then heated in tubes in an oven, and the melting points determined. It was found that all the samples of urea resin with and without flameproofing agents fused below 220°c. Melamine resins with and without flameproofing agents did not fuse at 265°c., the maximum temperature of the oven. Further tests at higher temperatures have shown that melamine resins containing flameproofing agents do not fuse, but char to hard masses.

These results may explain the behaviour of nylon net treated with resins and flameproofing agents. When nylon nets treated with methylated hydroxymethylmelamine, methylated hydroxymethylurea, or methylated hydroxymethylurea with the addition of thiourea are tested for inflammability by the vertical burning test [8], they all fuse and the melt drops off in molten masses which burn if the net is not flameproof. Nylon net treated with methylated hydroxymethylmelamine with the addition of thiourea (or other flameproofing agent) burns, leaving a hard carbonised skeleton, which retains the shape of the original net.

EFFECT OF CHROME DYES ON THE RESPONSE OF NYLON TO HEAT

If, as has been suggested, nylon dyed with chrome dyes burns by virtue of a restraining effect by the chromium on the dropping away of the nylon melt, this should be observable in one or both of two ways—

(*a*) The melting point of nylon should be raised by dyeing with chrome dyes

(*b*) The viscosity of the nylon melt should be increased by dyeing with chrome dyes

(*c*) A further possibility, if neither of these effects is observed, is that the presence of chromium causes a fall in the temperature of ignition of nylon.

(*a*) Samples of undyed, chromed only, and dyed-and-chromed nylon were inserted in tubes, and the melting points determined. No differences in melting point were observed.

(*b*) Since our object was merely to obtain an indication of whether there is a gross difference in viscosity between molten undyed nylon and nylon dyed with chrome dyes, it was felt that a simple arrangement might give the required result. Two short lengths of tube were marked at 1-cm. intervals with a file. The tubes were inclined at 45°, and 0·2 g. each inserted of undyed nylon and nylon dyed with a chrome dye and afterchromed. The tubes were placed in a furnace, the aperture of which was covered with a glass plate into which a glass tube had been sealed. The furnace was filled with nitrogen gas which was passed in through the tube. The temperature was raised to 340–350°c. and maintained at this level until the molten nylon had run down each tube. Frothing proved to be a source of difficulty, but it was possible to measure the rate of advance of the nylon. The large differences between flow times of different sets of results (Table VI) are presumably due to temperature differences which result from the use of a common laboratory muffle.

An attempt was also made to measure the viscosity at 425–450°c., but the low viscosity at this temperature made it extremely difficult to compare differences by this crude method.

(*c*) The possibility that the ignition temperature is lowered was investigated by heating nylon in a stream of oxygen, as bottled oxygen happened to be more readily available than compressed air. The apparatus used consisted of a Kjeldahl flask and a bung, into which were inserted a short inlet tube, a long tube reaching to the bottom of the flask, and a thermometer, the bulb of which was level with the end of the long tube. Oxygen was bubbled through sulphuric acid and into the flask through the short tube. The long tube served as an outlet for the gas. A small plug of nylon was inserted in the long tube, and the flask and contents were placed in the furnace with the long tube horizontal.

The temperature was raised slowly in the initial tests. It was found during these tests that the amount of nylon and the rate of flow of oxygen affected the results. The amount of nylon had to be such that the rate of gas flow through the tube was not reduced when the nylon melted. It was also found to be necessary to use a clean tube each time, as the presence of residue from a previous test on nylon dyed with chrome dyes depressed the temperature at which subsequent samples ignited. The presence of this residue from nylon dyed with chrome dyes also caused undyed nylon to behave like chrome-dyed nylon. Once a tube was contaminated, it proved to be practically impossible to clean it sufficiently thoroughly not to lower the ignition temperature of undyed nylon. The results obtained by this method (Table VII)

TABLE VI

Effect of Dyeing with Chrome Dyes on the Viscosity of Molten Nylon

Treatment	Set 1	Set 2	Set 3
	(Time taken to flow 3 cm.)		
Untreated nylon	7 min. 47 sec.	6 min. 22 sec.	5 min. 20 sec.
Chrome-dyed nylon	7 min. 55 sec.	6 min. 35 sec.	5 min. 20 sec.

were that chromed nylon and dyed chromed nylon ignited with a sharp crack at a definite temperature. Undyed nylon did not ignite.

TABLE VII

Effects of Chroming and Dyeing on Ignition Temperature

Treatment	Temperature of Ignition (°C.)		
Chromed only	480	490	491
Dyed and chromed ...	475	475	475
Untreated	Did not ignite at 575°c.		

The tests were repeated altering the conditions so that the untreated sample would also ignite (Table VIII). This was done by raising the temperature rapidly and by increasing the size of the sample. It had been noted that untreated nylon would ignite if the sample were sufficiently large. A repeat was done using bottled air instead of oxygen.

TABLE VIII

Treatment	Temperature of Ignition (°c.)					
OXYGEN						
Dyed and chromed ...	468	465	470	470		
Untreated	525	525	527	532		
AIR						
Dyed and chromed ...	516	521	504	513	518	513
Untreated	Did not ignite					

It was also found that thiourea does not affect the ignition temperature of chrome-dyed nylon.

Chromium is not unique in its action on nylon. Copper and iron affect the burning properties and the ignition temperature in a similar manner.

Discussion

NYLON DYED WITH CHROME DYES

It has been shown that the melting point and the viscosity of the melt of nylon are not altered by dyeing with chrome dyes, but the temperature of ignition is lowered. It has also been shown that the melting point of nylon is lowered by thiourea, but the temperature of ignition is not affected. If the difference between the melting point of nylon and its ignition temperature is the explanation for the fact that untreated nylon does not burn, it seems reasonable to suppose that modification in burning properties caused by the fall in ignition temperature can be countered by a corresponding fall in the melting point.

It is suggested that this may be the mechanism of the flameproofing of nylon dyed with chrome dyes by acid-forming flameproofing agents.

Thiourea is not a useful flameproofing agent for nylon dyed with chrome dyes because of its lack of wash fastness. It is possible to use resins incorporating thiourea, but this entails stiffening the material, and for this reason such a process would not be generally applicable.

NYLON FINISHED WITH THERMOSETTING RESINS

Acid-forming flameproofing agents cause melamine resin to char without fusion, and this may explain the failure of flameproofing agents to be effective in the presence of melamine and may also explain the observed behaviour of nylon net finished with melamine resins and a flameproofing agent. When nylon net finished with melamine resin burns, a molten mass is formed which drops off the burning nylon and continues to burn when falling. When flameproofing agents are added, no molten mass is formed. The advancing front of the flame leaves behind it a brittle charred skeleton, which retains the shape of the original net.

The melting point of urea resins is not lowered by flameproofing agents, and this excludes the possibility that a flameproofing agent acts other than on nylon alone. Since acid-forming flameproofing agents reduce the viscosity of the melt of nylon, it is suggested that this reduction in the viscosity allows the melt to drop away more readily, thus preventing flame propagation.

Appendix

TERYLENE

Terylene like nylon recedes from a flame without igniting when in the raw state, and becomes inflammable when finished with resins. The urea–thiourea resin finishes which give flameproof finishes on nylon likewise give flameproof finishes on Terylene, and melamine–thiourea resin finishes do not. The adhesion of the urea–thiourea finish to Terylene is too poor for it to have any practical application. Terylene net has to be given a stiff finish with melamine resins, and in consequence is inflammable. A few tests were made on Terylene to find whether the same principles apply as to nylon.

INFLUENCE OF FLAMEPROOFING AGENTS ON THE MELTING POINT OF TERYLENE

Samples of Terylene were prepared as in the case of nylon, with the results in Table IX.

TABLE IX

Effects of Flameproofing Agents on the Melting Point of Terylene

Flameproofing Agent	Melting Point (°c.)		
	Dry	Dry	Wet
Blank	253+	255–260	—
$NH_4H_2PO_4$ ...	247–253	250–255	215–225
NH_4Br	247–253	245–250	215–225
$(NH_4)_2SO_4$ ...	253+	250–255	215–225
NH_4CNS ...	233–240	230–235	215–225
$CS(NH_2)_2$ ...	233–240	235–240	215–225

The melting point of Terylene is therefore lowered by flameproofing agents in the same way as is the melting point of nylon. The action of thiourea on Terylene can be observed on Terylene braid, which burns in its untreated state. When a flame is applied to a Terylene braid into which

thiourea has been dried, it is observed that the braid does not burn because of the fluid nature of the melt, which drops off much more readily than from untreated braid.

* * *

The author would like to thank Mr. L. S. Laskowski, who carried out most of the experimental work, and the Council and the Director of Research of the Lace Research Association for permission to publish this paper.

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(*Received 19th June 1956*)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Assessment of Change in Colour—Use of Grey Scales and Colorimeter

In the past few years much progress has been made in the field of standardisation in the textile industry, and there is now a great deal of activity in the correlation of national standards, and the preparation of standards to be used internationally. At the same time increasing use is being made of instrumental aids for assessing properties about which there is a certain degree of subjectivity. This activity is particularly strong in the field of colour and colour-change assessment, and the data given below may be of use to those concerned in improving our techniques in this direction.

We have recently concluded a series of tests which entailed estimation, by means of the International Geometric Grey Scale, of the change of colour of a large number of dyed wool patterns. Many of these assessments were carried out also in other laboratories, and we have therefore had an excellent opportunity of judging the usefulness of the scale as a means of correlating the assessments made independently in more than one laboratory. In addition, the visual assessments were compared with values obtained on a commercial photometer, and in this way consistency in making assessments was checked.

The tests were carried out on dyed all-wool patterns supplied by several dyemaking companies, the range of colour being covered as far as possible with single-component dyes. Samples were subjected to one of the processes used for treating to Dylan shrink-resist standards, and S.D.C. No. 2 washing tests were carried out both on these and on untreated patterns. Assessments were then made of the change in colour on treatment and on washing the treated and untreated samples. The assessments were carried out not only in the Dylan technical service laboratory at Ambergate, but also in the laboratories of three of the dye companies concerned.

The numbers of patterns examined and the percentages of those for which there was found a difference of one grade on the Grey Scale between the assessments made by the Dylan technical service (D) and in the laboratories of the dye manufacturers (A–C) are given in Table I.

TABLE I

Assessments made by D and—	No. of Samples assessed	Percentage showing Difference of 1 unit
A	590	7
B	350	20
C	210	4 (1 unit or more)

For the first two companies none of the assessments differed by more than 1 Grey-scale unit from those found at Ambergate. In the case of the third dye company a few differed by 1½ units, but it is not worthwhile considering these separately.

This result constitutes remarkably good agreement between quite independent observers, and illustrates the importance of the Grey Scale as a practical reference standard.

An alternative to the use of visual assessments graded according to the spacings of the Grey Scale would be a photometric instrument that would be completely free from human error. It was thought that advantage should be taken of the large number of patterns available in order to compare assessments made visually and those made with a measuring instrument. The instrument available was the Color-Eye marketed by Instrument Development Laboratories Inc. of Massachusetts, U.S.A.

The machine is essentially a flicker photometer having three primary filters (X, Y, and Z) and provision for artificial light and daylight illuminations. This instrument measures the amount of light reflected by a sample as compared with a standard. In these experiments the standards were, for example, the untreated or unwashed samples, and these were compared with the treated or washed in just the same way as they are visually, using the Grey Scale. The difference between the two samples is expressed as a percentage reflectance

through each of the filters, and in order to compare the visual and instrument assessments it was first necessary to calibrate the instrument against the Grey-scale patterns.

This was done by making accurate visual matchings of the five Grey-scale patterns on plain-weave cotton fabric. Four experienced observers agreed that the finished dyeings were exact matches of the Grey-scale patterns. The dyeings representing steps 2, 3, and 4 on the Grey Scale were then compared with that representing No. 5, and the following scale of values (Table II) was found.

TABLE II

Number	Percentage thin when measured through the Neutral Filter
4	9
3	21
2	40

These readings are quite reasonable and in conformity with the theory of the scale.

After a number of trials with the Color-Eye, the following procedure was finally chosen for the examination of the dyed samples. The sample, e.g. treated, was compared with the standard, e.g. untreated, and readings were taken with each filter (X, Y, and Z). The sample and the standard were then rearranged in their respective holders and further readings taken, three or four sets of readings being taken altogether. In each set of readings either that figure (X, Y, or Z) showing the greatest deviation from 100 or the difference between the two extreme figures was taken, whichever was the greater. The greatest figures for the three or four sets were then averaged. The average reading was converted to the Grey-scale assessment by means of this table.

Average Deviation (%)	Grey-scale Rating
0–2	5
3–6	4–5
7–11	4
12–17	3–4
18–24	3
25–32	2–3

When this method was first used, the Color-Eye assessments were compared with those of a panel of observers, but as work progressed it was found possible to use the Color-Eye and one observer. Only in cases where the two failed to agree were further observers necessary, i.e. when the difference in assessment was one unit or more. Table III shows the number of occasions on which there was a large difference between visual and instrument assessments.

TABLE III

Comparison of Loss of Depth or Change of Hue	No. of Assessments	Proportion (%) showing difference of 1 unit	Proportion (%) showing difference of > 1 unit
On treatment of pattern	432	12	3
On washing untreated pattern	486	15	5
On washing treated pattern	486	23	9

The visual assessments were made on the basis of change of depth or hue, whichever was the greater. In all cases where there was a difference of more than 1 unit between the first visual and instrumental assessments, other visual assessments were found to agree with that first made, and the difference between the samples was seen to be one of hue. This observation also applied to a large number of the samples where a difference of one unit was found between the Color-Eye and the human observer.

In general, it was found that Color-Eye and visual observations agreed where differences of depth only were concerned, but that agreement was poorer if much change in hue occurred. With this reservation, the Color-Eye can be of the greatest assistance where many samples are to be compared. Conversely, the almost complete agreement within half a point between instrumental and visual Grey-scale assessments in the absence of hue differences emphasises the utility of the scale in maintaining consistency in the judgment of colour changes.

G. C. GIBBONS
G. E. KEY

STEVENSONS (DYERS) LTD.
AMBERGATE

5th April 1957

ERRATUM

Reports on the Progress of Applied Chemistry, Volume XL 1955 (J.S.D.C., 73, 168 (April 1957))—The price should be **60s. 0d.** (**40s. 0d.** to S.C.I. members).

Notes

Meetings of Council and Committees
May

Council— 3rd

Publications— 21st

Colour Index Editorial Panel— 13th and 22nd

Perkin Executive— 24th

Fastness Tests Co-ordinating— 10th

Terms and Definitions— 24th

Symposium— 29th

Deaths

We regret to report the loss by death of Mr. D. Cuthbertson and Mr. S. C. Lockwood.

Production of Dyes in Greece

A report from the British Embassy in Athens says that there are eight manufacturers of dyes in Greece, but only three— Biochrom Aniline Dye Industry, Chromatourgeia Athenon, and Chromatourgeia Metaxa— are of any size. They all produce mainly sulphur dyes. The total Greek production of organic dyes in 1954 was 280 tons, in which year 95·9 tons were imported, of which 60–70% went to the textile industry.

Production of Dyes in Rumania

Dyes, the production of which has recently been started in Rumania, include Direct Black RW, Direct Bright Orange, and Sulphocyanine GR and 5R, and additional yellow and brown dyes will be made shortly. The range of Carbon Blacks has been extended.

Light Fastness of Car Upholstery

A strongly worded editorial in the December 1956 issue of *Teinture et Apprêts* (No. 38, p. 253) states that certain manufacturers of motorcars continue to employ a mercury-vapour lamp for testing the light fastness of coloured textiles supplied to them. Since this may give misleading results, the journal offers to publish letters from the big car firms informing the general public that they have never used, or will renounce the use of, such a lamp for this purpose.

International Fastness Label *Felisol*

The International Association for the Fastness Label *Felisol* states that the following are eligible for membership (cf. J.S.D.C., **72**, 354 (1956)): (*a*) manufacturers of dyes which conform to the specifications laid down by the Association and (*b*) one national organisation of finishers in each country, incorporated for the purpose of promoting the aims of the Association, whose members are entitled to use the certification mark in the manner stipulated by the regulations. Membership has been granted to the following organisations: (*a*) BASF, FBy, CFM, DuP, FH, G, ICI, Fran, ACNA, Ciba, DH, Gy, S, Rohner, Saronio, and Vondelingenplaat; and (*b*) Vereniging van de Nederlandse Echtheidsmerk Textielgoederen (Arnhem), Associazione Italiana per il Marchio di Solidità *Felisol* (Milan), and Schweizerisches Markenpatronat *Felisol* (Zürich).

Discussions are in progress with other dye-making firms and with countries in which new national organisations of finishers are to be founded. Further information is obtainable from the Association at Zürich, Postfach 39, Switzerland.

Carrieur—le Langage pur

The French have recently been showing concern at the contamination of their language by English and American technical terms, so that, e.g. they have not only *des dancings* and *des skatings* but also *le cracking*. Although the mistranslation of English terms by their apparent French equivalents can certainly give rise to confusion, as with *control* and *contrôle* in international affairs, or *decade* and *décade* (ten days), it is to be hoped that the French purists will not emulate those Germans who would have replaced *orientieren* by "gemorgenländern" and *Lokomotiv* by "Zieh". Now the Comité Français de la Détergence has set up a Commission de Terminologie, which has been seeking a French equivalent for "carrier". After long deliberations the Commission has recommended the rejection of "chromoteur" as an etymological barbarism and the retention of *carrieur*, for it claims the English word to be merely an old French word with a slightly different spelling. However, a correspondent (*Teinture et Apprêts*, No. 39, p. 27 (Feb. 1957)) suggests that "carrieur" is practically unknown in practice, but that *véhiculeur* is already well established. Apparently, no-one has yet considered the obvious *porteur* for this purpose!

French Textile Terms

The Institut Textile de France is publishing a glossary of about 700 textile terms based on the second edition of *Textile Terms and Definitions*, published by the Textile Institute in 1955 (cf. J.S.D.C., **71**, 186 (1955)), which contains definitions put forward by the Society's Terms and Definitions Committee. An alphabetical list of French terms with their English equivalents appears in the *Bulletin de l'Institut Textile de France* (No. 65, pp. 79–101 (Feb. 1957)). The list of terms with definitions translated from the English versions will be spread over several future issues of the *Bulletin*. Before final publication of a separate pamphlet, careful consideration will be given to any comments received on the above lists.

Courpak Cellulose Acetate Film

Courtaulds Ltd. has adopted the trade mark *Courpak* for this product, which is marketed by British Cellophane Ltd.

New Books and Publications

Veredlerjahrbuch— Deutscher Färberkalender 1957

Edited by O. Mecheels. Pp. iv + 415 + 64 + 60. Munich: Franz Eder Verlag. Price, DM 12.00.

The 1957 issue of this annual publication follows the usual pattern of a series of reviews, some of them carrying useful literature references, on the development of fibres, dyes, and processing methods. Among the subjects covered are the bleaching of polyamide, polyester, and acrylonitrile fibres; the dyeing of wool at low temperatures; the coloration of materials with pigments and a binding agent; the application of azoic, vat, and solubilised vat dyes; the dyeing of cellulose triacetate and polyacrylonitrile fibres, with possibly the greater emphasis on the latter fibre.

The relative importance of finishing is emphasised by the number of articles dealing with the finishing of synthetic-fibre materials as well as of wool.

Innovations of interest are articles on dye constitution, on the problems of the changeover from mechanism to automation, and the inclusion of I.S.O. Tentative and Recommended tests.

The useful lists of new auxiliaries, detergents, and German fibres are included as usual.

This is a most useful and handy book, which fully maintains the high standard which we have come to expect of this publication.

A. Thomson

Acridines

By R. M. Acheson. Pp. xii + 409. New York and London: Interscience Publishers. 1956. Price, $12.50.

The volumes of this series— *The Chemistry of Heterocyclic Compounds*— have been published at irregular intervals since 1950, and although each is limited to a small field, the detailed and comprehensive presentation leads to substantial textbooks. This ninth member maintains the high standard of its predecessors, but the subject-matter has already been collected in book form (A. Albert, *The Acridines*, 1951).

It would be expected that the more recent book would summarise valuable later work, and the author claims to have included papers in the more important journals up to June 1954. In fact, however, there are indeed few references beyond 1950 outside the author's own published contributions to the field.

The problem of numbering the acridine ring system is first considered, since this has led to great confusion because of the prevalent use of two systems, one put forward by Graebe and the other by M. M. Richter. This difficulty can be appreciated by reference to the author's publication on the bromination of acridine (*J.C.S.*, 4142 (1954)), which gives 3-bromo- and 3:7-dibromoacridine as the products, numbering on the Richter system, but these are referred to in the book as the 2- and 2:7-compounds, since the Graebe system is adopted throughout.

The book brings out the importance of work on natural products, especially alkaloids, and other compounds showing well defined antibacterial activity including the widely known acriflavine. Before the introduction of paludrine, acridine antimalarials, especially mepacrine, were of considerable value.

Simple acridine dyes which colour fabrics yellow-green to red-brown are described, but they are unimportant, since the incorporated amino groups make them comparatively unstable to light and oxidising agents. A number of complex acridines, in contrast, have a wider range of colours and great value as vat dyes. With complex polycyclic structures it is often possible to trace out an acridine or acridone structure in compounds not ordinarily regarded as of acridine type, e.g. in flavanthrone. Many are of a so called "anthraquinone–acridone" class, a misleading term (not recommended in this book) to cover dyes of napthaquinone–acridone type (precisely described as *5:8:14(13)-naphth[2,3c]acridinetrione*). There are many such derivatives amongst the well known Indanthren dyes, which show a wide range of hues, and the preparation, structure, and value of many such types are given.

The chapter on visible and ultraviolet absorption spectra of acridine and its derivatives is specially prepared by L. E. Orgel.

Altogether an interesting book, well produced (printed in the Netherlands) and justifying the claim to reflect the importance and the interrelations of the various compounds covered by the title.

G. R. Ramage

Das Entfernen von Flecken aus Geweben (Kunstdetachieren)

By L. Jenko, E. Kopf, and E. Scheder. 5th edition 1957. Pp. 238. Munich: Franz Eder Verlag. Price, DM 22.00.

This guide to stain removing for cleaners begins with a brief description of dry cleaning in general and a consideration of the properties and relative merits of the solvents used. There follows an introduction to the techniques used by spotters in dry-cleaning plants. This section includes descriptions of the properties and uses of stain-removing and bleaching chemicals, together with illustrations of apparatus used for such techniques. The subsequent treatment of textile fibres and dyes is somewhat cursory, but probably adequate for a book of this nature.

171 stains of very different types are then described in some detail, together with practical suggestions for their removal. It is obvious that the latter are based on experience, and a name index to the stains gives quick access to the notes concerning any particular type. There appear to be no revolutionary suggestions, but the collection is very comprehensive. On testing the book as a work of reference, 9 out of 10 less common stains were found to be present in the text. The missing

item was chewing-gum stains, but this is perhaps excusable, since Vienna is now unoccupied.

A serious omission, however, is the failure to stress the value of charging the solvents with water, and in view of the success already attained by the new controlled systems of water incorporation, this is rather unfortunate. The effects of the methods in decreasing a number of articles requiring hand spotting are very properly the concern of this book, especially since a number of water-soluble stains are all given their respective niches in the text. To some extent the omission may be connected with the very high charges which some Continental cleaners command, a return which obviously allows for individual treatment of stains. By the same token, the length of some of the methods quoted for stain removal implies a slower production rate than that obtaining in this country.

A second criticism is of the sketchy way in which the identification of stains is detailed. Whilst it is impossible for a cleaner to carry out a lengthy identification of all troublesome stains, it is unhelpful to give methods of stain removal without a fairly comprehensive guide to stain characterisation. In many cases the book outlines the physical appearance and some of the properties of the stains, but when presented with an unknown stain the operator would require to work through the text until the right example appeared. It would not be too difficult to arrange some form of tabulation to eliminate a major part of this searching, thereby greatly adding to the value of the book to the working cleaner.

The volume is attractively and robustly produced, and the stain treatments hold none of the unconscious humour which comes from the salts-of-sorrel school in our household magazines. The authors obviously know their business, and since this book was introduced in 1931, and has subsequently run to five editions, there is clear proof that it is fulfilling a constant need. The German should not be found too difficult to those engaged in the cleaning trade, because of the similarity of the technical terms used.

S. R. Cockett

Synthetic Polypeptides

Preparation, Structure, and Properties

By C. H. Bamford, A. Elliott, and W. E. Hanby. Pp. xiii + 445. New York: Academic Press Inc. 1956. Price, $10.00.

The importance of proteins cannot be overemphasised. They are part of the very stuff of life itself. However, they are extremely complex materials and difficult to study, so that it is natural to use "model" substances to help elucidate their properties. The synthetic polypeptides provide such "models", and it is the purpose of the book under review to summarise and assess the work which has so far been done with these compounds. It would be difficult to find more suitable authors for such a book as this than Bamford, Elliott, and Hanby, for they, and their colleagues at the Courtaulds laboratories, have provided much of the impetus to the study of these hitherto rather neglected substances. The book is very much a record of their achievements, as they have made notable advances in all aspects of this subject. It is also a tribute to an enlightened industrial concern, which has developed such fundamental research without thinking of immediate financial return.

The plan of the book is to deal in separate sections with all the various facets of the work on synthetic polypeptides. After an introductory chapter, in which the relation of synthetic polypeptides to proteins is noted, there follows in Chapter II an account of the methods available for their synthesis. Chapter III describes physicochemical work on the mechanism of the important synthesis using *N*-carboxy-α-amino acid anhydrides. The possible configurations adopted by the polypeptide chains are fully discussed in Chapter IV, followed in Chapters V and VI by a description of infrared spectroscopic studies, especially those using polarised radiation, as applied to polypeptides. Chapters VII–IX give an account of X-ray diffraction studies. The general properties of the polypeptides, including a short account of their dyeing properties, are set out in Chapter X. Their biological properties are discussed briefly in Chapter XI, whilst Chapter XII relates them to the fibrous proteins, e.g. wool and silk. An appendix deals with some mathematical points, such as the optical transform in X-ray studies. References are listed at the end of each chapter, and a full subject index is given at the end of the book.

This book illustrates admirably how much of modern research is a team effort, specialists in different physical or chemical fields using their particular techniques in a study of one central problem, in this case the synthetic polypeptides. It is obvious that in a book of some 400 pages, dealing with such a wide range of methods and techniques, some compression and lack of clarity must result. Nevertheless, the references to the original work are very full, and the authors are to be congratulated not only for their presentation of the subject but also for finding and taking the opportunity to write this timely volume.

The American publishers have, as usual, made an extremely good job of the printing and the appearance of the book. It can be recommended to all who have interests in the polymer field.

G. J. Weston

Asbestos

from Rock to Fabric

By C. Z. Carroll-Porczynski. Pp. xv + 400. Manchester: The Textile Institute. 1956. Price, 55s. 0d.

This is the second book on inorganic fibres to appear in a relatively short period and indicates that the need for more published information on these fibres is realised and is in process of being met. The first book (*Anorganische Textilfaserstoffe*—vide J.S.D.C., **73**, 27 (Jan. 1957)) by reason of its recent appearance inevitably evokes comparisons

with the present volume, not the least striking of which is that the former was the work of a team, whilst *Asbestos* has been produced by a single author, who is thus to be complimented on his industry.

The book is also more easily readable than the German work, language difficulties apart, and should perhaps be considered as descriptive rather than as the work of reference which the latter undoubtedly is. In parts— for example the section on the industrial uses of asbestos— the book becomes absorbingly interesting, which is high praise indeed for any technical writing.

Commencing with the history and development of asbestos, three chapters on the occurrence, mining, and winning of the raw material follow. Interposed slightly out of sequence are the chemical and physical properties of the fibre. The processers of asbestos will find a large section devoted to textile manufacture, whilst, as noted above, consumers will find that part of the book devoted to application to be wide in scope and interest. Substitutes, glass fibres, testing methods are all covered, and a feature of the work is the lists of firms engaged in various activities connected with asbestos itself.

A considerable amount of information has been compressed into this book, the acknowledgments alone indicating the numerous firms and authorities consulted. Here and there, however, the zeal of the author for completeness has led to the inclusion of material not strictly connected with the work, apart from possible future competition with asbestos. Heat-resistant materials such as fluorocarbons and silicone rubber are more fully and more properly dealt with in other works. In the case of heat-resistant organic fibres, the connection is particularly thinly drawn and is even incomplete in that no reference is made to acetylated cotton. Credit must be given for the information on blends of asbestos with organic fibres. This last short section would merit expansion, for here the inorganic and organic fibres are on common ground.

The book is excellently produced and modestly priced in relation to the information given. Both the author and the Textile Institute are to be congratulated on their venture.

S. R. Cockett

Technique of Organic Chemistry
Volume III
Part II— Laboratory Engineering

Edited by A. Weissberger. 2nd revised edition 1957. Pp. ix + 391. London and New York: Interscience Publishers. Price, $8.00.

The first edition of this volume was published in 1950 and reviewed in this *Journal* in the same year (**66**, 547). The second edition is published in two parts, and Part I was reviewed earlier this year (J.S.D.C., **73**, 213 (May 1957)). In the new edition, the material has been arranged in a more systematic manner, and the text has been expanded considerably by the addition of new material. Part II of Volume III is devoted to non-diffusional subjects under the general heading *Laboratory Engineering*. The topics covered include— (i) Selection of Materials for the Construction of Equipment, (ii) Heating and Cooling, (iii) Grinding, Screening, and Classifying, (iv) Mixing, and (v) Operations with Gases. Of these, only Heating and Cooling, and Mixing, appeared as sections in the first edition. In each case the text has been fully revised, and new material added, to bring the work up to date. However, the main advantage of the new volume lies in the completely new sections, and of these undoubtedly the most needed, and most useful, is the section devoted to materials of construction. In general, most of the equipment used in the laboratory is made of glass, and use of other materials is avoided— even if they have distinct advantages over the usual glass apparatus. Much of this is due to a general ignorance of the properties of metals, plastics, and other materials under experimental conditions. The new chapter gives a very good introduction to the selection of materials for particular duties and contains nearly 150 references to original work. It is particularly welcome to see so much space devoted to the properties of plastics and non-metals.

Both the other new sections contain much useful information and combine to made a very valuable text, but it is unfortunate that in the chapter on Operations with Gases too much appears to have been attempted. The topics covered range from high-vacuum technique to storage of gas cylinders, and this leads to a general weakness of the text. A more systematic arrangement under less general headings would undoubtedly add to the value of this section.

This volume completes the second edition of Volume III and is a worthy companion to Part I. It is in effect a completely new book, since only a small portion of the work appeared in the first edition. It should prove very valuable to industrial chemists, especially in scaling up operations from bench to pilot plant.

P. J. King

New Books received

Acridines. R. M. Acheson. New York: Interscience Publishers Inc. 1956. Pp. xii + 409. $12.50.

1956 *Technical Manual and Year Book of the American Association of Textile Chemists and Colorists*. Volume **32**. W. H. Cady (editor). New York: Howes Publishing Co. Inc. 1956. Pp. 626. $6.50.

Symposium on pH Measurement. Philadelphia: American Society for Testing Materials. 1957. Pp. v + 101. $2.50.

Symposium on Steam Quality. A.S.T.M. Joint Research Committee on Boiler Feedwater Studies. Philadelphia: American Society for Testing Materials. 1957. Pp. v + 49. $1.75.

Farbmetrik. Hans Arens. Berlin: Akademie-Verlag. 2nd edition 1956. Pp. xi + 83. DM 13.80.

British Standard Glossary of Leather Terms. London: British Standards Institution. 1956. Pp. 24. 5s. 0d.

Asbestos from Rock to Fabric. C. Z. Carroll-Porczynski. Manchester: Textile Institute. 1956. Pp. xv + 400. 55s. 0d.

The Encyclopedia of Chemistry. G. L. Clark (editor-in-chief). New York: Reinhold Publishing Corpn. 1957. Pp. xvi + 1037. 156s. 0d.

Proceedings of the International Wool Textile Research Conference. Australia 1955. Volumes **A**, **B**, **C** (Parts I and II), **D**, and **E** (Parts I and II). Melbourne: Commonwealth Scientific and Industrial Research Organisation. 1956. Pp.— (**A** & **B**), 28 + 302, (**C**) 1–256 and 257–507, (**D**) 272, (**E**) 1–314 and 315–620.

Statistical Methods in Research and Production with special reference to the Chemical Industry. O. L. Davies (editor). London: Oliver & Boyd (for Imperial Chemical Industries Ltd.). 3rd edition 1957. Pp. xii + 396. 45s. 0d.

Progrès réalisés dans l'Application des Matières Colorantes. Volume 2. L. Diserens. Paris: Éditions Teintex. 2nd edition 1952. Pp. viii + viii + 533.

1957 Year Book. Philadelphia: Federation of Paint and Varnish Production Clubs. Pp. 168. $2.50.

Chemie und physikalische Chemie der Textilhilfsmittel, Band 2. H. Frotscher. Berlin: VEB Verlag Technik. 1955. Pp. 174. DM 15.00.

Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents 1888–1956. J. C. Harris and R. Bernstein. Philadelphia: American Society for Testing Materials. 1956. Pp. iii + 39. $1.50.

Das Entfernen von Flecken aus Geweben (Kunstdetachieren). L. Jenko, E. Kopf, and E. Scheder. Munich: Franz Eder Verlag. 5th edition 1957. Pp. 238. DM 22.0.

Chemical Engineering Reports— How to search the Literature and prepare a Report. K. A. Kobe. New York: Interscience Publishers Inc. 4th edition 1957. Pp. viii + 175. $3.00.

Das Farbensehen. S. W. Krawkow. Berlin: Akademie-Verlag. 1955. Pp. xi + 168. DM 16.00.

Die wissenschaftlichen Grundlagen der Trocknungstechnik. O. Krischer. Berlin: Springer-Verlag. 1956. Pp. xx + 400. DM 46.50.

Veredler-Jahrbuch— Deutscher Färberkalender für das Jahr 1957. 61 Jahrgang. O. Mecheels (editor). Munich: Franz Eder Verlag. iv + 64 + 1 + 415 + 64 + 60. DM 12.

Jahrbuch der Textilveredlung. Band 3. G. Meier. Berlin: VEB Verlag Technik. 1956. Pp. 932.

Fusion Methods in Chemical Microscopy. W. C. McCrone Jr. New York: Interscience Publishers Inc. 1957. Pp. vii + 307. $6.75.

The Chemistry and Technology of Leather. Volume 1— *Preparation for Tannage.* F. O'Flaherty, W. T. Roddy, and R. M. Lollar (editors). New York: Reinhold Publishing Corpn. 1956. Pp. xiii + 495. 112s. 0d.

International Discussion of Problems in Color Metrics. (Heidelberg, 1955). M. Richter (editor). Göttingen: Musterschmidt Verlag. 1956. Pp. 113–328. DM 23.40.

Künstliche organische Farbstoffe und ihre Anwendungen. W. Seidenfaden. Stuttgart: Ferdinand Enke Verlag. 1957. Pp. vii + 256. DM 44.50.

Review of Textile Progress. Volume 7. 1955. Manchester and Bradford: Textile Institute and Society of Dyers and Colourists. 1956. Pp. 581. 42s. 0d.

Official Methods of Analysis (1957) of the Society of Leather Trades' Chemists. Croydon: Society of Leather Trades' Chemists. 3rd edition 1957. Pp. vii + 200. 18s. 0d.

Tafeln zur Prüfung des Farbensinnes. K. Velhagen Jr. (editor). Stuttgart: Georg Thieme Verlag. 21st edition 1952. Pp. 15 + 19 (37 colour plates). DM 18.40.

Elementary Practical Organic Chemistry. Part 1— *Small Scale Preparations.* A. I. Vogel. London: Longmans, Green & Co. Ltd. 1957. Pp. xv + 347 + xiv. 21s. 0d.

Technique of Organic Chemistry. Volume 3. Part II— *Laboratory Engineering.* A. Weissberger (editor). New York: Interscience Publishers Inc. 2nd edition 1957. Pp. ix + 391. $8.00.

Resonance in Organic Chemistry. G. W. Wheland. New York: John Wiley & Sons Inc. 1955. Pp. xiii + 846. 120s. 0d.

Neuere Erkenntnisse über das Trocknen von Textilien. W. Ziemba. Switzerland: Zollikofer & Co. A.G. 1955. Pp. 67.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Evaluation of the Usefulness of Conditioning Ovens in relation to the Correctness of their Working

W. Szmelter

Zesz. Nauk. Politech. Lódz., No. 13 (*Włókiennictwo* 2), 37–58 (Sept. 1956)

A detailed analysis of the working of several types of standard textile-conditioning apparatus is made. A theoretically ideal example of such an instrument is discussed. Several constructional aspects are enumerated, which when applied in practice would, according to the author, lead to standardisation of the conditioning of textiles. T.Z.W.

PATENTS

Padding Machines

J. Dungler *BP* 763,654

In a pad suitable for pad dyeing the trough is formed by end plates engaging the ends of the rolls. These plates extend above the rolls. There are side walls whose vertical edges engage the end plates and whose lower edges engage the surfaces of the roller. C.O.C.

Storing Cloth during Continuous Treatment

J. Dungler *BP* 768,876

When cloth is being treated continuously in open width it can be stored during treatment without imparting tension to it by passing it around a series of parallel guide rolls. The number, arrangement and the size of the rolls are so chosen that the cloth can continuously pass successively several times around the rolls in such a way that it forms during passage over some of the rolls, outer layers superimposed on the previous layer and, on the other rolls, inner layers beneath the previous layer. The sum of the lengths of the arcs embraced by the cloth on the rolls on which it forms outer layers being equal to the sum of the lengths of the arcs embraced by the cloth on the rolls on which it forms inner layers. All the rolls have the same diameter and the arcs embraced by the cloth around the different rolls have the same angular extent. The cloth passes around the successive rolls alternately forming an upper outer layer and an under inner layer. C.O.C.

Machine for Shrinking Tricot Fabrics

I. Olsson *BP* 766,513

The fabric is led through a chamber in which it is treated with wet saturated steam. Each pair of rollers that carry the fabric through the chamber rotate less quickly than the preceding pair, the amount of this decrease in rotation depending on the degree of shrinkage required.

C.O.C.

Controlling the Tension in Cloth, Paper, Plastics, etc. during Dyeing, Printing or Finishing

Research Corpn. *BP* 769,134

Discharging Static Electricity from Textiles
Institut Textile de France *BP* 769,055

Apparatus is described in which a stream of gas previously exposed to a radio-active substance is projected against the charged material. This causes complete and instantaneous discharge of the static electricity in the material. C.O.C.

Injection of Liquids into Sheet Material
Facsite S.P.A. *BP* 772,376

Sheets of paper or board, etc., are passed between two cylinders, that are forced together, one or both of the cylinders being coated with a resilient material containing parallel rows of mutually staggered individual recesses. The recesses are continually filled with liquid which is transferred to the sheet under compression and penetrates deeply into it. Excess liquid is removed by passing the sheet between two heated cylinders. K.W.

II— WATER AND EFFLUENTS

Properties and Applications of Sequestering Agents
R. L. Smith

Chem. and Ind., 1284–1291 (10 Nov. 1956)

A review, particularly of ethylenediamine- and hydroxyethyl-ethylenediamine-acetic acids. Properties dealt with include stability of chelates, competition in chelation equilibria, sequestration of iron under alkaline conditions, and also industrial applications. Mention is made of the use of sequestering agents in thirteen different industrial applications, including the textile, paper, and leather industries. A.J.

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Redox Potential of Dichromate
B. V. Ptitsyn and V. F. Petrov

J. Gen. Chem. U.S.S.R., **26**, 3233–3239 (Dec. 1956)

The reaction—

$$Cr_2O_7^{2-} + 14H^+ + 6e \rightleftharpoons 2Cr^{3+} + 7H_2O$$

does not describe the true thermodynamic equilibrium of the reduction process. An intermediate valency stage, probably that of quinquevalent Cr, must be considered. For identical pH values the redox potential of dichromate is found to decrease in the series:

$$HClO_4 > H_2SO_4 > HNO_3 > CH_3COOH.$$

G.J.K.

PATENTS

Ungelatinised Tertiary Amino Alkyl Ethers of Amylaceous Materials— Size
National Starch Products *BP* 765,880

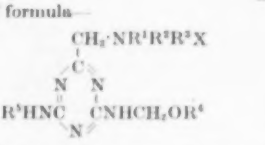
Starch is treated in an alkaline medium with an etherifying agent containing a tertiary amino radical, e.g. β-dimethylamino*iso*propylchloridehydrochloride, under conditions that avoid gelatinisation of the starch, giving products of formula

$$X\text{-}O\text{-}R^1\text{-}\overset{R^2}{\underset{}{N}}\text{-}R^3$$

(X = starch radical; R^1 = alkylene or hydroxyalkylene; R^2 and R^3 = Alk or Ar but may not both be Ar; or R^1 + R^2 may form a saturated ring with the N atom), e.g. starch $\cdot O\cdot CH(CH_2)CH_2\cdot N(CH_3)_2HCl$. The products may be used to increase the strength of paper, as thickening agents in the plisse process for printing nylon fabrics, or as adhesives. K.W.

Antistatic Agents
Rohm & Haas *BP* 763,323

Compounds of formula—



(R^1 and R^2 = Alk or together form a saturated divalent aliphatic chain which forms a 5- or 6-membered ring with the N atom; R^3 = Alk, alkenyl or aralkyl; X = anion or together with R^3 = $-CH_2CH_2COO-$; R^4 = H or Alk of 5 C; R^5 = H or R^4OCH_2) e.g. dimethylaminoacetoguanamine, have affinity for textile fibres to which they impart an antistatic finish which has good fastness to washing. They do not form resins on the fibre. C.O.C.

Antistatic Agents
Boehme Fettchemie *BP* 769,039

The neutral product of the addition of an alkylene oxide to an acid ester of a phosphoric acid and a hydroxyl compound of > 5 C, used in aqueous solution or dispersion is a good antistatic agent, e.g. the condensate of 2 mol. C_{15}–C_{18} fatty alcohols, 1 mol. P_2O_5 and 4–6 mol. ethylene oxide. C.O.C.

Antistatic Agent
Owens-Corning Fiberglas Corpn. *USP* 2,731,367

Treatment of all kinds of fibres both inorganic and organic with a water-soluble polysiloxanolate having one or more saturated alkyl, aryl or mixed alk-aryl groups having relatively short C chains attached directly to Si, e.g. potassium *iso*butyl polysiloxanolate, gives them an antistatic finish fast to washing and dry cleaning. C.O.C.

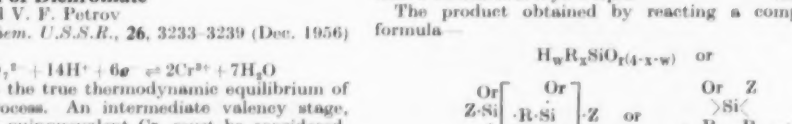
Catalysts for Resin Finishes
Boehme Fettchemie *BP* 767,249

Salts of ammonia or organic bases with high molecular organic sulphonic or phosphonic acids are excellent condensation catalysts for use with resin finishes. Thus spun viscose rayon fabric is padded with its own wt. of a liquor containing 250 g. per l. of 60% urea formaldehyde precondensate and 10 g. of an ammonium salt of an acid ester of sulphuric acid with alcohols of 12–18 C, dried at 70°c. and baked for 5 min. at 130°c. The angle of recovery of the fabric rises from 54° to 132° by this treatment and it has a soft and warm handle. C.O.C.

Titanated Alkoxysilanes—Waterproofing Agents
Allied Chemical & Dye Corpn. *BP* 766,810

The product obtained by reacting a compound of formula—

$$H_wR_xSiO_{r(4-x-w)} \quad \text{or}$$



or $RSi(Or)_mO_{\frac{3-m}{2}}$

(R = organic groups of 1–18 C; r = aliphatic groups of 1–18 C; w = 0 or 1; x = 0–3; x + w < 4; z = 1 or 2; Z = 2 monovalent group of same class as R or O; m = ½–2) with a Ti salt of HCl, HBr or a saturated fatty acid of 1–4 C. Thus ethyl orthosilicate (1 mol.) is mixed with $TiCl_4$ (0·25 mol.). The mixture rapidly warms exothermally to 75°c. after which heat is applied externally and the mixture brought to 200°c. in 2 hr. It is neutralised with Na dissolved in ethyl alcohol and decolorised with carbon. After vacuum distilling to remove material boiling up to 160°c./10 mm. Hg an ethoxy-silyl titanate which is a viscous amber oil is obtained. Such products are strongly absorbed on polar surfaces of paper, textiles, etc. to which they impart excellent water-repellency with no loss in strength. C.O.C.

Fixation of Mechanical Finishes
Ciba *BP* 763,268

Water-soluble alkyl ethers of urea-HCHO condensate (> 2 mol. HCHO to 1 mol. urea) are excellent agents for use in fixing mechanical finishes.

BP 763,465

The solution also contains an aluminium salt. C.O.C.

Polysiloxane Water-repellent Compositions
Midland Silicones *BP* 766,864

Compositions containing (1) 5–75% by wt. of a liquid or hydrocarbon solvent-soluble polysiloxane having on average 0·05–1·0 silicon-bonded H atom per Si atom, the remainder of the valencies of the Si atoms being satisfied by O atoms, aliphatic hydrocarbons of < 6 C and on average 0–0·1 Ar per Si atom, there being on average 1·6–2·5 hydro-

carbon radicals and H atoms per Si atom, and (2) 25–95% by wt. of a hydroxylated polysiloxane of viscosity < 1,000,000 cs. at 25°c. and having on average 1·4–2·0 monovalent hydrocarbon radicals per Si atom, applied to textiles impart a water-repellent finish which is resistant to grease spotting. Thus a 2% solution in xylene of a mixture of 40% of a hydroxy end-blocked dimethyl polysiloxane of viscosity 129 cs. at 25°c. and 60% of trimethyl end-blocked methyl hydrogen polysiloxane of viscosity 25 cs. at 25°c. applied to wool gaberdine so that the fabric absorbed 1·6% by wt. of siloxane and then drying the fabric and baking for 10 min. at 300°F. imparted an excellent water-repellent effect free from mark-off and grease spotting. The fabric had greatly increased resistance to shrinking on laundering. C.O.C.

Trichloronitropropanol as Solvent for Acrylonitrile Polymers
Chemstrand Corpn. *USP* 2,732,359

Trichloronitropropanol is an excellent solvent for polymers containing ⩽ 70% acrylonitrile. It is normally a solid and so has to be used at above its m.p. (45–50°c.) and preferably at 65–100°c. The resulting solutions can be cooled to room temperature without gelling or precipitation of the polymer. When such solutions are extruded into water the trichloronitropropanol is only slowly dissolved and so the coagulated product is clear, transparent and free from voids. C.O.C.

IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Production and World Commerce in Colouring Matters
E. A. Sack
Teintex, **22**, 173–179 (March 1957)

Seven tables are given, based on O.E.E.C. and national data, showing the production in metric tons in 1954 and 1955, values in U.S.A. dollars per kilogram, and exports and imports in metric tons and French francs. S.R.C.

Ionising Radiation as a "Catalyst" for Chlorination and Bromination
R. A. Cox and A. J. Swallow
Chem. and Ind., 1277–1278 (3 Nov. 1956)

Irradiation, under anaerobic conditions, of a suspension of *N*-bromosuccinimide in a mixture of toluene and carbon tetrachloride with fast electrons from a 2-MeV. van der Graaff generator, to a total dosage of ca. 10^{21}eV., leads to the formation of benzyl chloride in 69% yield. In general, radiation-induced halogenations lead to the same products as those produced by free-radical catalysts. A.J.

Preparation of α-Alkylnaphthalenes
S. I. Sergievskaya and T. S. Safonova
J. Gen. Chem. U.S.S.R., **26**, 3479–3485 (Dec. 1956)

Reactivity of the Methyl Group. XIV— Effect of a 1- or 2-Naphthoyl Group
L. Chardonnens and H. Thomann
Helv. chim. Acta, **39**, 1892–1899 (Dec. 1956)

Friedel–Crafts reaction between 4-methyl-3-nitrobenzoyl chloride and naphthalene (also its 1-chloro or 1-methyl deriv.) gives *o*-nitrotoluenes substituted by 1- and 2-naphthoyl residues. These react with benzaldehydes or *p*-nitrosodimethylaniline. Thus naphthoyl groups exert an activating effect comparable with that of a benzoyl or a phenylsulphonyl substituent. Hydrolysis of the azamethins leads to *o*-nitroaldehydes, which yield 6:6′-naphthoylindigos by reaction with acetone and sodium hydroxide. H.E.N.

Chloromethylation of Anthracene
E. U. Yu. Gudrinietse and G. Ya. Vanag
J. Gen. Chem. U.S.S.R., **26**, 3123–3125 (Nov. 1956)

Bisulphite Compounds of 4-Nitroso-1-naphthol and of 1-Naphthol
S. V. Bogdanov and N. N. Karandasheva
J. Gen. Chem. U.S.S.R., **26**, 3365–3368 (Dec. 1956)

α-Naphthol derivatives react with bisulphite to give the tetralonesulphonate, and this reacts with hydroxylamine to give the corresponding oxime. G.J.K.

Reversibility of Aromatic Nitration
P. H. Gore
J.C.S., 1437–1439 (March 1957)

Examples of acid-catalysed migrations of aromatic nitro groups recently recorded by Paussacker and Seroggie (cf. *Chem. and Ind.*, 1290 (1954); *J.C.S.*, 1897 (1955)) are discussed, and an alternative mechanism is suggested which indicates that aromatic nitration may be reversible, although it is generally understood to be irreversible. In support, data are reported from the action of H_2SO_4 on 9-nitroanthracene which indicate reversible nitration to be possible. It is emphasised, however, that normal aromatic nitration should continue to be regarded as essentially irreversible. H.H.H.

Nitration of 4-chloro-2-nitrobenzoic Acid
H. Goldstein and E. Schaaf
Helv. chim. Acta, **40**, 369–372 (March 1957)

Reaction of Nitrous Acid with Salicylic Acid in Acetic Acid
A. A. Nemodruk
J. Gen. Chem. U.S.S.R., **26**, 3283–3285 (Dec. 1956)

In excess HNO_2 diazotised *o*-aminophenol can be prepared (88% yield), thus offering distinct advantages over other methods. *o*-Nitrosophenol is shown to be formed as an intermediate. G.J.K.

Reduction of Azo Compounds with Hydrazine
W. H. Stafford, M. Los, and N. Thomson
Chem. and Ind., 1277 (3 Nov. 1956)

Azo compounds are reduced by reaction with excess of hydrazine. The reaction may be catalysed by Raney nickel, porous tile, or adventitious impurities. Azo compounds containing no electron-releasing substituents are reduced only to the hydrazo stage. If electron-releasing groups are present, reduction is to the two amines, except for hydroxy-azo compounds, with which pure products cannot be isolated. A.J.

Thermal Rearrangement of Aromatic Hydrazo Compounds
M. Vecera, J. Gasparič, and J. Petránek
Chem. and Ind., 299 (9 March 1957)

It is suggested that the main stage in the thermal rearrangement of hydrazo compounds of the benzene series is homolytic cleavage of the N–N bond to form two radicals with mesomeric shift of electrons toward the *ortho* and *para* positions. Semidine types are the main products. W.R.M.

Reaction of Free Radicals in Solution. VI— Mechanism of the Decomposition of Alkyl Aryl Triazens in presence of Water and Acids
V. Ya. Andakushkin, B. A. Dolgoplosk, and I. I. Radchenko
J. Gen. Chem. U.S.S.R., **26**, 2972–2980 (Nov. 1956)

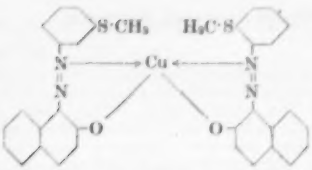
The decomposition of these compounds in aqueous and organic media, both in presence and in absence of organic acids, is investigated, and mechanisms based on the intermediate formation of free radicals are given. G.J.K.

Syntheses with Complex Compounds. III— Preparation of Azo Compounds from Chromotropic Acid and from other 1:8-Dihydroxynaphthalenesulphonic Acids
V. I. Kuznetsov and A. A. Nemodruk
J. Gen. Chem. U.S.S.R., **26**, 3285–3290 (Dec. 1956)

The use of Ca salts of chromotropic acid in the coupling with aromatic diazo compounds markedly decreases the oxidative side-reaction, thus increasing the yield of the azo dye. The preparation of some new mono- and dis-azo dyes from chromotropic acid is given. G.J.K.

Internal-complex Salts of Azo Compounds. IV— Reaction of Copper Salts with Certain *o*-Hydroxy- and *o*-Amino-*o*′-methylthioazo Compounds
V. I. Mur
J. Gen. Chem. U.S.S.R., **26**, 3208–3212 (Nov. 1956)

A large number of Cu chelates, e.g.—



are prepared from the azo compounds obtained by diazotisation of 2-aminothioanisole and coupling with β-naphthol, 1-phenyl-3-methyl-5-pyrazolone, β-naphthylamine, and 1-phenyl-3-methyl-5-aminopyrazole. Two forms of the Cu chelates are identified (1 : 1 and 1 : 2 Cu : dye ratio) except with the *o*-amino derivative, which yields 2-(*o*-methyl-thiophenyl)-naphtho-1′ : 2′-4 : 5-triazole. Prolonged heating of 1-*o*-methylthiophenylazo-2-naphthol with $CuSO_4$ and pyridine yields the Cu salt of *o*-di-(2-hydroxy-naphthalene-1-azo)phenyl sulphide (ratio 1 : 1). G.J.K.

Spectra and Halochromism. III— Reaction of Parafuchsine, Aniline Blue, and their Carbinols with Acids

V. F. Lavrushin and T. M. Shmaeva

J. Gen. Chem. U.S.S.R., **26**, 3075–3081 (Nov. 1956)

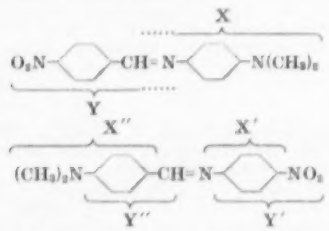
The authors establish the limiting concn. of strong acids at which the acids do not react with the auxochromic groups of Aniline Blue. A conjugated carbonium structure is suggested for the above-mentioned triphenylmethane dyes, the carbonium ion being claimed to be the chromophore. G.J.K.

Structural Effects on Spectra. I— Comparative Study of the Colour exhibited by Azamethins of the Type $NO_2{\cdot}C_6H_4{\cdot}CH{:}N{\cdot}C_6H_4{\cdot}R$ and $R{\cdot}C_6H_4{\cdot}CH{:}N{\cdot}C_6H_4{:}NO_2$— Reflection and Absorption Spectra

V. A. Izmail'skii and E. A. Smirnov

J. Gen. Chem. U.S.S.R., **26**, 3042–3060 (Nov. 1956)

The spectra, both in solution and in the solid state, of the above derivatives are studied. The effect of substituents (R = H, OH, $N(CH_3)_2$) is investigated, and the bathochromic shifts produced by these substituents are shown to be based upon the electron-donating and electron-accepting character of the individual parts (X and Y) of the complex chromophoric system, e.g.—



The spectral curves are given and the spectral similarity of *p*-nitroaniline to benzylidene-*p*-nitroaniline and of *p*-dimethylaminobenzylideneaniline to *p*-nitroaniline and benzylidenenitroaniline are discussed. G.J.K.

Influence of Acids and Alkalis on the Absorption Spectra of "Pyridine" Dyes

N. E. Grigor'eva, I. K. Gintse, and A. P. Severina

J. Gen. Chem. U.S.S.R., **26**, 3096–3104 (Nov. 1956)

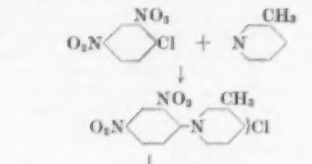
The authors investigate the absorption spectra of the precursors of cyanine dyes of the structure R·NH·CH:CH·CH:CH·CH:N·R in 96% alcohol with varying amounts of hydrochloric, excess carbonic, and glacial acetic acids and of caustic soda, in both the visible and the ultraviolet regions. The degree of hydrolysis of the dye salt (depending upon the basicity of the cation) is correlated with the bathochromic displacement. The equilibrium in alcoholic solution of dyes having electron-donating substituents is shifted by alkali towards the base, whereas alcoholic solutions of dyes having electron-accepting substituents are converted by excess alkali into deeply coloured anionic dyes. G.J.K.

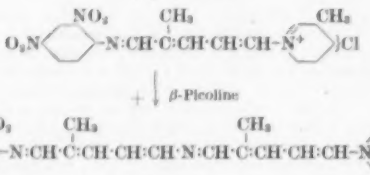
Reaction Products of β-Picoline with 2:4-Dinitrochlorobenzene

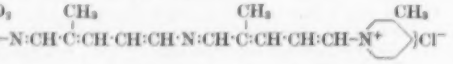
N. E. Grigor'eva, I. K. Gintse, and N. G. Karpyuk

J. Gen. Chem. U.S.S.R., **26**, 3455–3462 (Dec. 1956)

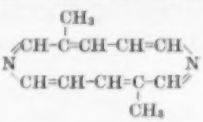
The following products are obtained depending on the amount of β-picoline present—







The end-product, an unstable violet polymethin dye, decomposes to give 2:4-dinitrochlorobenzene, β-picoline, and a brown residue whose formula is suggested to be—



G.J.K.

Azamethin Dyes. III— Azamethin Dyes from 3-Alkyl-1-(carboxy- and dicarboxy-2-benzothiazolyl)-5-pyrazolones

I. A. Solov'eva, M. V. Krasheninnikova, and G. I. Arbuzov (and in part A. G. Guseva)

J. Gen. Chem. U.S.S.R., **26**, 3036–3042 (Nov. 1956)

Azamethin dyes from the condensation of *p*-nitrosodiethylaniline and mono- and di-carboxylic acids of benzothiazolyl-5-pyrazolones are described. The presence of carboxyl groups in the benzothiazole ring causes a slight bathochromic displacement. Use of these dyes in gelatin film causes a hypsochromic displacement of the absorption band relative to alcoholic solutions. The spectra of 1-aryl- and 1-benzothiazolyl-pyrazolones are compared. G.J.K.

Direct Amination of Benzothiazoles

R. P. Vel'tman

J. Gen. Chem. U.S.S.R., **26**, 3388–3391 (Dec. 1956)

Synthesis of Phenothiazines. I— Synthesis of Dialkylaminoacyl-2-chlorophenothiazines

S. V. Zhuravlev and A. N. Gritsenko

J. Gen. Chem. U.S.S.R., **26**, 3385–3388 (Dec. 1956)

Synthesis of Halogenophenazines. V— Iodophenazines

V. P. Chernetskii and A. I. Kiprianov

J. Gen. Chem. U.S.S.R., **26**, 3465–3467 (Dec. 1956)

Recent Developments in Anthraquinonoid Acid and Direct Dyes

J. Günthard

Amer. Dyestuff Rep., **46**, 9–21 (14 Jan. 1957)

Translation of paper in *Textil-Rund.*, **11**, 639–644 (Oct. 1956)— cf. J.S.D.C., **73**, 106 (March 1957).

Fluorenacenes and Fluorenaphenes. Syntheses in the Indenofluorene Series. V— Methyl Derivatives of *cis*-Fluorenacene

L. Chardonnens and M. Schmitz

Helv. chim. Acta, **39**, 1981–1985 (Dec. 1956)

Synthesis of Chlorinated Copper Phthalocyanines

V. F. Borodkin, Yu. G. Erÿkalov, and K. V. Usacheva

J. Appl. Chem. U.S.S.R., **29**, 1606–1608 (Oct. 1956)

When 72 g. of tetrachlorophthalic anhydride reacts with 100 g. of urea, 13·5 g. of Cu_2Cl_2, and 3 g. of ammonium

molybdate as a catalyst, hexadecachlorophthalocyanine in 56–60% yield is produced. The reaction is carried out at 180–230°C. in 900–1000 ml. of diluent, e.g. nitrobenzene or trichlorobenzene, for 10–25 hr. The reaction product, after washing with solvent and water, is dried, and then treated with 10% HCl soln. and 10% NaOH soln., washed till neutral, and dried at 80–90°C. Direct chlorination gives a better product. T.Z.W.

Stability and Kinetics in Complex Formation. VII— Tetraphenylporphin Complexes
S. Fallab and H. Erlenmeyer
Helv. chim. Acta, **39**, 2130–2132 (Dec. 1956)

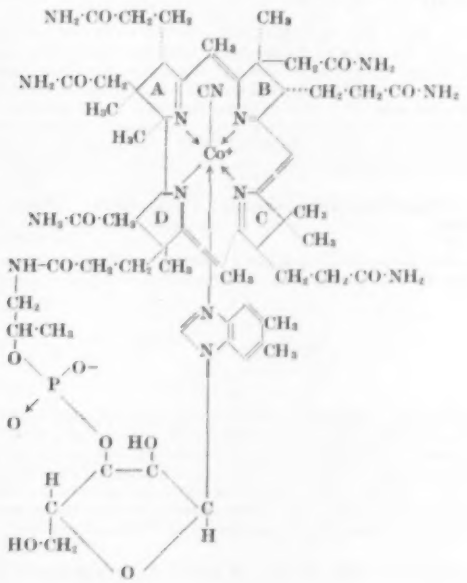
Carotenoid Syntheses
O. Isler, H. Lindlar, M. Montavon, R. Rüegg, G. Saucy, and P. Zeller

VII— Total Synthesis of Zeaxanthene and Physalien
Helv. chim. Acta, **39**, 2041–2053 (Dec. 1956)

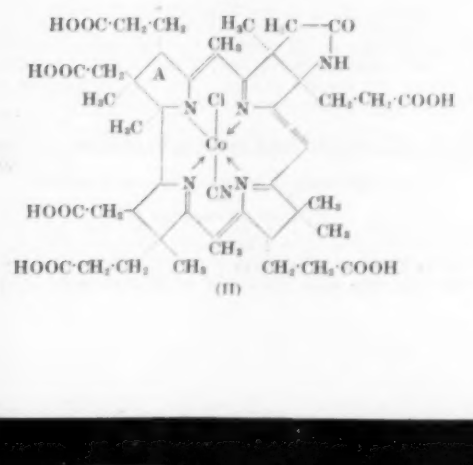
VIII— Total Synthesis of Kryptoxanthene and a Further Synthesis of Zeaxanthene
Ibid., **40**, 456–467 (March 1957)

Chemistry of the Vitamin B_{12} Group. V— Structure of the Chromophoric Grouping
R. Bonnett, J. R. Cannon, V. M. Clark, A. W. Johnson, L. F. J. Parker, E. L. Smith, and Sir A. Todd
J.C.S., 1158–1168 (March 1957)

Structures for vitamin B_{12} (I) and its hexacarboxylic acid degradation product (II) (cf. *ibid.*, pp. 1148–1158) containing six rather than five conjugated double bonds are adopted, and reactions bearing on this point are considered. Mild alkali in the presence of oxygen cyclises the acetamide side-chain of ring B of I to give dehydrovitamin B_{12} (III), a γ-lactam, whereas the same side-chain is converted into a γ-lactone by an equiv. of a halogenating agent. More than one equiv. has a marked bathochromic effect on the chromophore, and III gives a cryst. monohalogeno deriv.; mechanisms for these cyclisations are discussed. H.H.H.



(I)



(II)

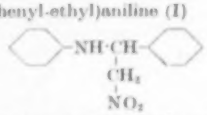
Effect of Degree of Dispersion on the Colour of Red Iron Oxide Pigment
L. N. Uspenskaya, A. Kh. Girenko, and M. S. Ostrikov
J. Appl. Chem. U.S.S.R., **29**, 1601–1604 (Oct. 1956)

The colour of the pigment depends upon the proportion of the constituent fractions. The best colour is produced by the fraction consisting of particles whose radius is 0·2–0·3 μ. T.Z.W.

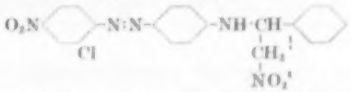
PATENTS

Monoazo Pigments and Disperse Dyes
FBy *BP* 771,566

N-(2-nitro-1-phenyl-ethyl)aniline (I)



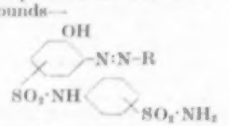
and its derivatives are coupled with suitable diazo compounds to give pigments and disperse dyes. Thus diazotised *o*-chloro-*p*-nitroaniline is coupled with I, prepared by addition of nitromethane to benzal-aniline or by addition of aniline to *ω*-nitrostyrene, to give



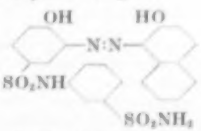
which dyes cellulose acetate orange and nylon red. E.S.

Metal(Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.
Ciba *BP* 771,309

The metal complexes from 1 atom of Co and 2 mol. of monoazo compounds—



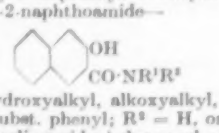
(R = residue of *ortho*-coupling naphthol, naphthylamine or keto-methylene compound) dye wool, nylon, etc., from neutral or weakly acid baths. Thus 2-chloronitrobenzene-5-sulphonyl chloride is condensed with metanilamide to give the corresponding *N*-*m*-sulphamylanilide. Replacement of the Cl atom by OH by means of an alkali or alkaline earth metal hydroxide, followed by reduction of the NO_2 group to NH_2 gives 2-aminophenol-4-sulphon-*N*-*m*-sulphamylanilide. This is diazotised and coupled with an alkaline soln. of β-naphthol to give



Heating with aq. $CoSO_4$ in presence of NaOH gives the complex, which dyes wool bordeaux. E.S.

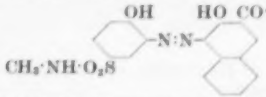
Metal-complex Monoazo Dyes for Wool, Nylon, etc., from 3:2-Hydroxynaphthoic Acid Amides
S *BP* 772,019

The 1:2 chromium and cobalt complexes of monoazo compounds obtained by coupling a diazotised *o*-aminophenol containing a sulphamyl or alkylsulphonyl group with a 3-hydroxy-2-naphthoamide—



(R^1 = H, Alk, hydroxyalkyl, alkoxyalkyl, aralkyl, *cyclo*alkyl, phenyl, a subst. phenyl; R^2 = H, or lower Alk; or NR^1R^2 = heterocyclic residue) dye wool, nylon, etc. red, brown, violet, blue, and green from neutral or weakly acid

baths. Thus 2-aminophenol-4-sulphon-*N*-methylamide is diazotised and coupled with 3-hydroxynaphthalene-2-carbo-*N*-methylamide under alkaline conditions to give

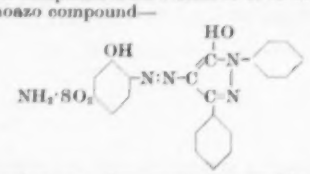


Stirring with $CoSO_4$ in presence of NaOH and tartaric acid at 60°C. gives the complex, which dyes wool and nylon bordeaux. E.S.

Metal (Chromium and Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

Ciba *BP* 771,318

The metal complexes from 1 atom of Cr or Co and 2 mol. of the monoazo compound—



dye wool, nylon, etc., from neutral or weakly acid baths. Thus 2-aminophenol-5-sulphonamide is diazotised and coupled with 1:3-diphenyl-5-pyrazolone and the monoazo compound so formed heated at 70–80°C. with aq. $CoSO_4$, or at the boil with aq. sodium potassium chromosalicylate to give respectively an orange-brown and a yellowish-red dye for wool. E.S.

Metal (Chromium and Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

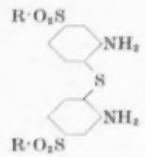
Ciba *BP* 771,320

The metal complexes from 1 atom of Cr or Co and a mixture of 2 mol. of metallisable monoazo compounds, one containing an SO_3H group and the other, no SO_3H or COOH group, but a sulphone or sulphonamide group, dye wool, nylon, etc. from neutral or weakly acid baths. Thus 1 mol. of each of the monoazo compounds 1-amino-2-naphthol-4-sulphonic acid→β-naphthol and 2-amino-4-chlorophenol-5-sulphon-*N*-methylamide→β-naphthol are boiled with aq. sodium chromosalicylate. The chromium complex so formed dyes wool blue. E.S.

Red Disazo Acid Dyes from Diaminodiphenyl Sulphide Derivatives

FBy *BP* 769,618

Derivatives of diaminodiphenyl sulphides—

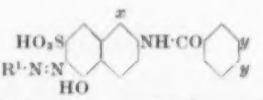


(R = Ar, Alk, amino, or subst. amino) are tetrazotised and acid-coupled with γ acid or an *N*-alkyl-γ acid to give red acid dyes of good light fastness. Thus 2 mol. of 4-chloro-3-nitrobenzenesulphondimethylamide are condensed with 1 mol. of an alkali metal sulphide, and the dinitro compound so formed is reduced with Fe to the required diamine (R = $N(CH_3)_2$), which is tetrazotised and coupled with 2 mol. of γ acid in presence of acetic acid. The product dyes wool reddish bordeaux from a weakly acid bath. E.S.

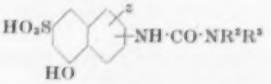
Metallisable Disazo Direct Dyes

S *BP* 771,331

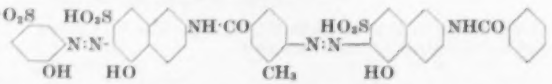
An extension of *BP* 687,328 (J.S.D.C., **69**, 128 (1953)) Aminomonoazo compounds—



(R^1 = aryl of benzene or naphthalene series having a substituent *ortho* to the azo link capable of metal-complex formation; *x* = H or SO_3H; one *y* = CH_3, the other *y* = NH_2) are diazotised and coupled with (*a*) aliphatic or heterocyclic keto-enol compounds, (*b*) an *ortho*-coupling phenol, naphthol, or naphtholsulphonic acid free from acylamino groups, (*c*) compounds—



(*z* = H, Cl, or SO_3H; R^2 = H or Alk; R^3 = H, Alk, *cyclo*alkyl, aralkyl, or aryl), or (*d*) if R^1 carries a sulphone group, the second coupling component may be an *N*-acylaminonaphtholsulphonic acid. The disazo dyes so formed are converted in substance or on cellulose fibres into copper and nickel complexes. Thus the monoazo compound 2-amino-4-methylsulphonylphenol→*N*-*p*-amino-*m*-methylbenzoyl-J acid is diazotised and coupled with *N*-benzoyl-J acid to give the disazo compound—

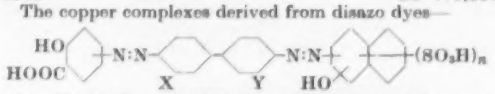


which dyes cellulose red by the aftercoppering process. E.S.

Metal (Copper)-complex Disazo Direct Dyes

CFM *BP* 771,330

The copper complexes derived from disazo dyes—

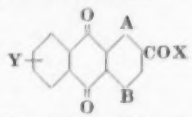


(X = H, O Alk, or COOH; Y = O Alk or COOH; *n* = 1, 2 or 3; the coupling components may contain further substituents) dye cellulose and nylon various hues from claret to grey. Thus tetrazotised dianisidine is coupled first with an alkaline soln. of 1 mol. salicylic acid and the intermediate product is then coupled with 1-amino-8-naphthol-4:8-disulphonic acid. Heating with ammoniacal copper sulphate gives the copper complex, which dyes cotton grey. E.S.

1:4-Diaminoanthraquinone Derivatives; Disperse Dyes

BASF *BP* 769,183

Blue disperse dyes of good fastness are obtained by acid reduction of compounds of formula—



(A and B = –NHOH, –NO or $-NO_2$; X = H, Alk or –OH; Y = H, Hal, Alk, –OH, $-NH_2$ or NO_2) which are made by treating anthraquinone-1(N):2-isoxazoles with nitrous or nitric acids. Thus, to 1:4-dinitro-anthraquinone-2-carboxylic acid in methanol at 40–50°C. are added conc. HCl and stannous chloride. After stirring 30 min. the product is filtered, washed with methanol and then water. E.T.

Sulphuric Acid Esters of leuco-Anthraquinone Vat Dyes

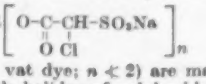
DH *BP* 769,973

A mixture of pyridine, diethyl*cyclo*hexylamine and/or one of the three isomeric diethyl-(monomethyl*cyclo*hexyl)-amines is used as the medium for treating the leuco-vat dye with chlorosulphonic acid. Other reaction media are given in *BP* 719,621 (J.S.D.C., **71**, 157 (1955)) and *BP* 745,365 and 745,368 (J.S.D.C., **72**, 394 (1956)). E.T.

Sulphochloracetic Acid Ester Salts of *leuco* Vat Dyes

FH *BP* 769,688

Water-soluble salts of *leuco* vat dyes of formula—



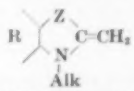
(R = residue of a vat dye; *n* < 2) are made by reacting *leuco* vat dyes with halides of sulphochloracetic acid in

presence of a tertiary base and converting the products into stable water-soluble salts. Thus, to 5:5′:7:7′-*tetra*bromindigo (2·5) mixed with pyridine (60) are added crude sulphochloracetic acid (7) and *p*-toluene sulphochloride (7·5) at 35–40°c. Fe (1) is added and the melt stirred at 45–50°c. whilst passing CO_2 through the mixture until a clear solution is obtained. After 3 hr. excess Fe is removed by filtering and the filtrate is added to cold water (300) and conc. H_2SO_4 (40). The product is filtered, washed and redissolved in water (150) and $NaHCO_3$ (1·5). After screening, the final product is obtained by salting and drying under reduced pressure. E.T.

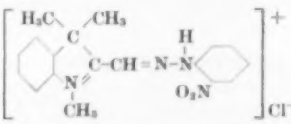
New Hydrazone Dyes for Polyacrylonitrile

Ciba *BP* 769,163

Dyes free from $-SO_3H$ and –COOH groups are prepared by combining the diazo-compound of a *mononitro-ortho*-aminobenzene or of a 1-aminoanthraquinone with a base of formula—



(R = benzene or naphthalene residue fused to a heterocyclic ring where Z = S or $>C(Alk)_2$). Also, the tetrazo compound of an *aa*-diaminoanthraquinone may be additively combined on both sides with such a base. Thus, a solution of diazotised 1-amino-2-nitrobenzene is added slowly at 0–5°c. to a solution of 1:3:3-trimethyl-2-methyleneindoline in HCl and ice water. The excess HCl is neutralised with Na acetate solution and after stirring 16 hr. at 0–5°c. is filtered and washed with ice water. The product—

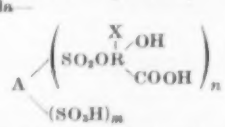


dyes polyacrylonitrile from an acetic acid bath a red-yellow of good light fastness. The dyes are usually sufficiently soluble for application from aqueous solution; otherwise they may be applied from dispersions. E.T.

Phthalocyanine Mordant Dyes

DH *BP* 768,444

Water-soluble esters of phthalocyanine *tetra*sulphonic acids of formula—



(A = subst. or unsubst. phthalocyanine residue with or without metal; the $-SO_3H$ and modified $-SO_3H$ groups are in the 3- or 4-positions of the phthalimide from which the phthalocyanine is made; m = 0–3; n = 1–4 and $m + n$ = 4; R = a benzene nucleus the –OH and –COOH *ortho* to one another; X = H, Hal, –OH, CH_3 or SO_3H) are made by condensing a phthalocyaninesulphonic acid chloride with a dihydroxybenzenecarboxylic acid. Thus, to a paste of crude copper phthalocyanine*tetra*sulphonic acid chloride mixed with ice is added a solution of 1:3-dihydroxybenzenecarboxylic acid and NaOH at 5–10°c. The dye is finally separated by addition of HCl and filtration. Redissolving in Na_2CO_3 soln. and salting gives a blue water-soluble powder. The product is an afterchrome dye yielding dyeings of good wet fastness and very good light fastness. E.T.

Carbon Black

Phillips Petroleum Co. *USP* 2,731,328

A method of producing good quality carbon black from low-grade carbonaceous materials, e.g. powdered coal, pitch, etc. C.O.C.

Titanium Dioxide

Chemical Foundation *USP* 2,732,310

A titanium ore is heated with chlorine in presence of a carbonaceous reducing agent. The $TiCl_4$ formed is purified and treated with aqueous nitric acid to yield rutile-type hydrated titanium dioxide and a gaseous mixture of Cl and nitrosyl chloride. The gases are recovered and separated—nitrosyl chloride converted into nitric acid and chlorine. By this means the pigment can be obtained either very pure or deposited on an extender. C.O.C.

Pigments from Dyes and Sulphonamide-melamine-formaldehyde Resins

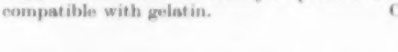
Switzer Bros. *BP* 769,344

The products obtained by incorporating a dye in a finely divided thermoplastic resinous co-condensate of an aromatic sulphonamide or a C-alkyl derivative thereof having > 1 reactive H atoms attached to N and a formaldehyde condensate of melamine or an alkyl derivative thereof and alkanol modified methylol melamines, are faster to light than other coloured pigments containing the same dye. Thus *o*- and *p*-toluenesulphonamide resins are heated with partly condensed unmodified melamine–formaldehyde resin and while molten C.I. Basic Red 1 and C.I. Basic Violet 10 added. On cooling and grinding a bluish-red pigment is obtained having strong fluorescence in daylight. C.O.C.

Non-diffusing Dyes

Eastman Kodak Co. *USP* 2,732,382

The products obtained by treating a triphenylmethane dye, azo dye, anthraquinone dye or other dyes containing a C-hydroxyalkyl or N-hydroxyalkyl group with a synthetic polymer containing carboxylic halide, carboxylic anhydride or similar groups which can esterify the hydroxyalkyl group of the dye have the same colour characteristics as the original dye and their diffusibility, e.g. in photographic emulsion, is low or absent. Thus the dye—



dissolved in pyridine and polyacrylyl chloride dissolved in acetone are refluxed together for 16 hr. to yield a product the sodium salt of which is readily dispersible with water and compatible with gelatin. C.O.C.

V— PAINTS; ENAMELS; INKS

PATENTS

Wet Ink Finishing Varnish

Sherwin-Williams Co. *USP* 2,732,355

A varnish for applying to wet prints on metal sheets, etc. to inhibit bleeding or spreading of the wet ink comprises an alkyd resin in a petroleum hydrocarbon solvent, and small amounts of a liquid halogenated aromatic hydrocarbon and a polymeric silicone, e.g. polymethyl silicone of viscosity 100 cs. C.O.C.

Metallic Paint

Engine Parts Manufacturing Co. *USP* 2,731,436

A metallic or bronzing lacquer which does not "sour" or "green and gel" and remains usable for prolonged periods comprises a metallic pigment, especially a Cu-Zn alloy, a poly(alkyl methacrylate) lacquer base dissolved in xylene, or benzene or toluene and methyl or ethyl alcohol, and difluorodichloromethane. C.O.C.

Coating Material Resistant to Deterioration and Chalking

General Motors Corpn. *USP* 2,731,354

Addition of a strong oxidising agent to paints, lacquers, etc. materially inhibits chalking, water-soluble permanganates and dichromates being particularly suitable for this purpose irrespective of the type of vehicle or pigment used. C.O.C.

Light-sensitive Pigmented or Dyed Resinous Compositions for Decorating Ceramics (IX p. 279)

VI— FIBRES; YARNS; FABRICS

Repeat Faults in Textiles

K. Vogler

Textil-Rund., **12**, 3, 115–123 (March 1957)

A number of manufacturing faults are discussed with suggestions for tracing them to their sources. S.R.C.

Physical Chemistry of Native Cellulose
H. U. Usmanov
J. Polymer Sci., **23**, 831–838 (Feb. 1957)

Cellulose appears in cotton tissue with the flower and pod. The synthesis of cellulose in cotton is at first very slow, and is accelerated by the 15th day after bloom, with a subsequent slowing and complete cessation on about the 40th day after bloom. The D.P. is at first very low, but shows an abrupt increase by the 25–30th day after bloom. The filament consists almost entirely of α-cellulose. Some properties of early cellulose are compared with those of more mature cellulose. W.R.M.

Kinetics of the Xanthation of Cellulose
B. Philipp

II— Influence of the Chemical and Morphological Structure of Alkali Cellulose
Faserforsch. und Textiltech., **8**, 45–53 (Feb. 1957)

Kinetics of fibre xanthation, using excess carbon disulphide, is investigated with respect to the effects of temperature, concentration, press factor, and morphological structure of the fibres.

III— Reaction of Alkali Cellulose with Mixtures of Carbonyl Sulphide and Carbon Disulphide
Ibid., **8**, 91–98 (March 1957)

The formation of mixed xanthates is investigated, and the behaviour of their solutions briefly described. The decrease in viscosity of viscose on addition of polyhydric alcohols to the steeping liquor is ascribed to the presence of carbonyl sulphide causing formation of a mixed xanthate. W.R.M.

Degradation during Storage of Viscose Rayon Staple
T. N. Kleinert, V. Moessmer, and A. Apuchtin
Textil-Rundsch., **12**, 124–129 (March 1957)

Lustrous and TiO_2–matt viscose rayons, with and without softening agents, were stored in the dark for long periods. At 20°C. insignificant changes in D.P. and tenacity, and no change in the degree of whiteness, occurred. On the other hand, with the thermally accelerated yellowing test there was a distinct reduction in the degree of whiteness and increase in caustic solubility and caustic light absorption. Considerable variations in results were possibly due to fibre impurities. Alkaline yellowing tests on hemicelluloses showed that the γ-cellulose portion of high pentosan content possessed a marked yellowing tendency. S.R.C.

Isolation of a Cuticle Sheath from Wool Fibres
R. L. Elliott and J. B. Roberts
J.S.D.C., **73**, 95–97 (March 1957)

A cuticular sheath, isolated from the wool fibre by oxidation with potassium dichromate followed by extraction with ammonia, consists of epicuticle and endocuticle. The material resistant to this treatment was examined in the optical microscope, and its morphological significance was confirmed by electron-microscopic studies of gold-shadowed residues. Electron microscopy also revealed a fibrillar and microfibrillar structure of the endocuticle, thus explaining the grooved appearance of the surface of damaged wool fibres (e.g. after wet chlorination). The sulphur content of the dichromate–ammonia-resistant endocuticle (comprising at most 2% of the original wool) is shown to be appreciably higher than that of the complete wool fibre. This sulphur is shown to be present in the form of cystine, but the resistance of the cystine in the cuticular layer to dichromate oxidation is unexplained.
AUTHORS

Bilateral Cortical Structure of Pakistani Carpet Wool
N. Ahmad and W. R. Lang
Text. Research J., **26**, 954–956 (Dec. 1956)

The cortical staining behaviour of 14 Pakistani carpet wools towards Methylene Blue is reported. In both medullated and non-medullated fibres orthocortex and paracortex are present in approx. equal proportions. Fibres were classified, on the basis of mean diameter, with regard to the type of cortical segmentation shown. All medullated fibres possess a central orthocortex surrounding the medulla. A central orthocortex is also common in coarser non-medullated fibres. The finer fibres exhibit a greater variety of segmentation types than do fine merino wools. J.C.F.

Structure of Keratin. II— Amino-acid Content of Fractions isolated from Oxidised Wool
C. Earland and C. S. Knight
Biochim. Biophys. Acta, **22**, 405–411 (Dec. 1956)

The hydrolysates of fractions isolated from wool oxidised with peracetic acid have been examined by paper chromatography. It has been shown that the chromatograms are more complex than those of untreated wool, and the fractions soluble and insoluble in ammonia solution contain lanthionine sulphone. The spot on the chromatogram corresponding to this substance is very weak and of equal intensity in the two fractions. No evidence for the presence of any other cross-links in the insoluble fraction could be found. It was shown that, in addition to oxidising all the cystine residues in wool, peracetic acid oxidised some of the lysine and arginine residues. Wool, after reaction with potassium cyanide solutions followed by peracetic acid, produces on hydrolysis lanthionine sulphone and cysteic acid. This shows that conversion of the cystine residues to lanthionine by cyanide solutions is not quantitative. P.G.M.

Regularities in the Chemical Structure of α-Keratins
R. D. B. Fraser and T. P. MacRae
Nature, **179**, 732–733 (6 April 1957)

X-Ray diagrams of wool have been obtained after treatment with iodine, osmium tetroxide, barium acetate, phosphotungstic acid, cupric acetate, and silver nitrate. It is found that wool can bind appreciable quantities of heavy-metal ions without significant effect on the X-ray diagram. Binding appears to be confined to non-crystalline regions. In the case of mercuric acetate the X-ray diagram is considerably modified, giving evidence of a regular three-dimensional distribution of chemical reactivity in the crystallites. W.R.M.

The α–β Transformation in Keratin
E. G. Bendit
Nature, **179**, 535 (9 March 1957)

The variations of peak intensity of the three prominent reflexions of the X-ray diagram of wool with extension suggest that at 20% extension the α–β transformation is well advanced and there is often no rapid change between 30% and 50% extension. Comparison of X-ray diagrams before and after extension showed no significant change in the intensity of the 5·1-A. and 9·8-A. reflexions and a marked increase in the intensity of the 4·65-A. reflexion. These observations are not in agreement with Astbury's hypothesis of a "molecular" α–β transformation. W.R.M.

Reaction of Silk Fibroin with Oxidising Agents
C. Earland and J. G. P. Stell
Biochim. Biophys. Acta, **23**, 97–102 (Jan. 1957)

After reaction with solutions of hypochlorite, acid and alkaline permanganate, chlorine dioxide, or iodine, silk fibroin becomes insoluble in the usual solvents. Chlorine in acid solution reacts but does not render fibroin insoluble. It is shown that the solubility of fibroin is highly dependent on the state of the tyrosine residues, and that fibroin may be rendered insoluble when the residues undergo oxidation or substitution reactions. P.G.M.

Fibre Formation from Concentrated Polymer Solutions
Y. Nishijima and G. Oster
J. Polymer Sci., **23**, 99–102 (Jan. 1957)

A theory of fibre formation from concentrated polymer solutions is presented. Above a certain concentration the polymer molecules lose their identity and the solution becomes a continuous network. Under these conditions the solution is spinnable. Calculations show that the drawn-out network which forms the fibre has long-range periodicity. W.R.M.

Polyester Fibres
P.-A. Koch
Textil-Rundsch., **12**, 131–138 (March 1957)

Continuing the series of fibre tables, the condensed information gives names of fibre, manufacturers, inventor, and developers, raw material production, constitution, properties, yarns available, and uses, together with 53 references. S.R.C.

Carboxyl and Hydroxyl Absorption in the Infrared Spectrum of Polyethylene Terephthalate

D. Patterson and I. M. Ward

Trans. Faraday Soc., **53**, 291–294 (March 1957)

Identification of the absorption bands due to alcoholic and carboxylic OH groups in the infrared spectrum of polyethylene terephthalate has been carried out by replacement of the terminal H atoms by deuterium. The band assigned to the carboxylic OH bond-stretching vibration has been separated from overlying bands. Measurements of the intensities of the bands associated with both types of end-group can be made, providing a possible method of estimating each. W.R.M.

X-Ray Studies of Fibres from Modified Aromatic Polyesters

B. Falkai and G. Bodor

Faserforsch. und Textiltech., **8**, 114–119 (March 1957)

Melting points, glass temperatures, the course of crystallisation, and dye absorption have been determined for fibres obtained from copolymers of ethylene terephthalate and pimelic, azelaic, and sebacic acids. These are affected to nearly the same extent by the different acids. Melting points, crystallinity, and glass temperatures decrease with increasing amount of acid. Light absorption of the amorphous polymers increases with increasing amounts of acid, but that of the polymers heated at 216°C. varies only slightly. W.R.M.

Colour Formation in Polyacrylonitrile

E. M. La Combe

J. Polymer Sci., **24**, 152–154 (March 1957)

Bases cause discoloration of polyacrylonitrile and acrylonitrile-containing polymers on heating. A reaction mechanism is suggested involving formation of closed rings containing conjugated carbon–nitrogen double bonds. W.R.M.

Acrylic Fibres. XVIII— Branched Polyacrylonitrile. I— Preparation of Branched Polyacrylonitriles with the aid of *NN'*-Methylenebisacrylamide

A. Hunyar and E. Roth

Faserforsch. und Textiltech., **8**, 99–108 (March 1957)

Polymerisation of acrylonitrile in which minute amounts of *NN'*-methylenebisacrylamide have been incorporated gives branched polyacrylonitriles. Variations of viscosity and D.P. with percentage branching are examined. W.R.M.

Properties of Concentrated (Spinning) Solutions of Acrylonitrile-Vinylidene Chloride Copolymers

Z. A. Zazulina, Z. A. Rogovin, and G. V. Vinogradov

Colloid J. U.S.S.R., **18**, 674–678 (Nov.–Dec. 1956)

Woven Polyvinylidene Chloride Monofil (Saran and I.G.G. Vestan)

J. Wrieth

Textil-Rund., **12**, 129–130 (March 1957)

Physical, textile, and chemical resistance data are given for I.G.G. Vestan together with some uses. S.R.C.

Isotactic Polymers

G. Natta

Chem. and Ind., 296–298 (9 March 1957)

Summary of lecture. By use of stereospecific catalysts it is possible to prepare polymers from α-olefins in which the substituents all have the same configuration relative to the main chain of the polymer. The highly crystalline nature of such isotactic polymers is briefly discussed. Stereospecific catalysts used are prepared by reaction of a transition-metal compound with an alkyl or hydride of a metal from Groups I–III of the Periodic System (Ziegler catalysts). Kinetic studies are briefly considered, and results are consistent with an anionic chain mechanism of polymerisation. Stereoblock polymers have been prepared from propylene. Isotactic polypropylene can be melt-spun into fibres with strength equivalent to those from nylon and Terylene. W.R.M.

PATENTS

Improving the Affinity for Dyes of Polyglycol Terephthalates

ICI *BP* 769,405

The products obtained by the melt blending of poly(glycol terephthalates) especially poly(ethyleneglycol terephthalate) with a polyester from *iso*phthalic acid and a glycol have greater affinity for disperse dyes than the unmodified poly(glycol terephthalate). C.O.C.

Trichloronitropropanol as Solvent for Acrylonitrile Polymers (III p. 271)
Cellulose–Methanol System (XI p. 283)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Home Laundering of Wash-and-wear Garments of Hydrophobic Fibres

G. D. Rawlings, H. E. Stanley, and P. R. Wilkinson

Text. Research J., **26**, 974–980 (Dec. 1956)

A study of the effect of fabric construction and laundering variables on the minimum-care properties of fabrics made from Dacron, Orlon, and nylon, and from blends of these fibres with each other and with cotton, rayon, and wool. For acceptable non-iron behaviour, it is suggested that garments be washed at 100–120°F., and the temperature then reduced to 60°F. by running in cold water whilst continuing agitation. Rinsing should be done at 60°F. Where a tumble dryer is available, fabrics may be washed at 120°F. and tumbled dry at 170°F. Measurements of the effort required to iron dress shirts showed that garments containing 100% or 65% Dacron were much easier to iron than cotton shirts. J.C.F.

Investigation of the Adsorption of Detergent and Builders on Cotton and Soil by Radiotracer Methods

T. F. Boyd and R. Bernstein

J. Amer. Oil Chem. Soc., **33**, 614–619 (Dec. 1956)

The results of studies on the adsorption of detergents and builders by cotton and soil are presented. Materials used included radioactive samples of dodecylbenzenesulphonate, sodium sulphate and carbonate, trisodium phosphate, tetrasodium pyrophosphate, and sodium tripolyphosphate. P.G.M.

Origins of Dry Cleaning

S. M. Edelstein

Amer. Dyestuff Rep., **46**, 1–8 (14 Jan. 1957)

Distribution of Hydrogen Peroxide between Water and Organic Solvents

F. R. Paulsen

Chem. and Ind., 1274 (3 Nov. 1956)

Measurements have been made of the partition of H_2O_2 between H_2O and 41 organic liquids, including esters, ethers, ketones, and alcohols. In all cases the value of the partition coefficient—

$$K = \frac{H_2O_2 \text{ in aq. layer}}{H_2O_2 \text{ in organic layer}}$$

depends on the concn. of H_2O_2 and increases with increasing temp. Within each series, K increases with increasing complexity of the organic compound. A.J.

Chlorite Bleaching using Organic Esters

J. Rosset and Y. Guyonnet

Teintex, **22**, 181–194 (March 1957)

Used batchwise with normal immersion times, esters cause less evolution of ClO_2 than acids, and the useless ClO_3^- and Cl^- contents are diminished. Standing baths are possible, and the white is equal to that obtained with acids. Application of ethyl tartrate as a continuous impregnation process reduces the steaming time and allows stains to be bleached without boiling. Corrosion is also greatly reduced. S.R.C.

PATENT

Bleaching of Protein Fibres

Wolsey *BP* 767,744

The fibres are treated with 1–40% of their wt. of a sulphur-containing reducing agent in aqueous solution at pH 2–7 and 0–100°C. and also with a fluorescent brightening agent before, during or after bleaching. Thus wool (10 g.) previously bleached with H_2O_2 was treated for 30 min. at 40°C. in a solution (300 c.c.) containing $NaHSO_3$ (2 g.), HCOOH (0·1 g.) and diaminostilbenedisulphonic acid (0·01 g.). On exposure to sunlight the wool had much better colour compared with a standard sample similarly treated in absence of $NaHSO_3$. C.O.C.

VIII— DYEING

Hydrogen Bond
C. A. Coulson

Research, **10**, 149–159 (April 1957)

Review article with 45 references. W.R.M.

Production of Multicolour Effects on Cloth
H. Faldik

Textil Praxis, **12**, 165–169 (Feb. 1957)

Various methods are described for the production of multicoloured effects in cloth composed of unions of wool and man-made fibres and also in all-wool materials by introducing chlorinated wool and wool treated with resists. Ombré and other special effects are also described. B.K.

Harmonising Colour Combinations
J. Lotichius

De Tex., **15**, 1554–1562 (Nov. 1956)

The factors involved in colour observation are reviewed, and five rules are formulated to determine whether a colour combination will harmonise. These include frequency of observation, clear contrast, light and dark colours of various hues in contrast, awareness of the human sensitivity to red, and the use of colours of the same colour group. E.M.

Developments in Phthalocyanines for Textiles
E. E. Bowker

Dyer, **117**, 355–360 (1 March 1957)

A review.

Measurements of Diffusion Coefficients of Azoic Coupling Components (2-Hydroxy-3-naphthanilides) in Cellulose
A. S. Dunn

J.S.D.C., **73**, 92–94 (March 1957)

A suitable apparatus is described for measuring the rapid rates of dyeing which occur, using thick cellulose model filaments. Diffusion coefficients and energies of activation for diffusion of six coupling components have been evaluated. Author

Influence of Hydrophilic Substances on the Diffusion of Direct Dyes into the Cellulosic Fibre
B. N. Mel'nikov and P. V. Moryganov

Colloid. J. U.S.S.R., **18**, 711–716 (Nov.–Dec. 1956)

It is found that in the case of two black direct dyes the addition of pyridine to the dyebath leads to increased diffusion coefficients within the fibre and decreased energy of activation, compared with results obtained in absence of pyridine under otherwise identical temp. and electrolyte concn. conditions. The effect of pyridine is greater the greater the dye affinity for the fibre, and the suggestion is made that the addition of hydrophilic substances may replace the use of dyeing temp. above 100°C. G.J.K.

Rate-of-dyeing Curves— I
S. Hafenrichter

Textil Praxis, **12**, 171–176 (Feb. 1957)

This represents a first report which deals with methods of determination and presentation of rate-of-dyeing curves together with the practical application of the results. Reference is made to a number of examples illustrating the effect of variation in temp. and time on the rates of exhaustion of vat, disperse, and metal-complex dyes. B.K.

Decomposition of Direct Dyes at High Temperatures
D. R. Sharma

Colourage, **3**, 17–20 and 45–48 (Oct. 1956)

The four direct dyes— Pyrazol Fast Red 8BL (8), Chloramine Blue 2B (8), Diphenyl Green 2B Supra (Gy), and Calcomine Black (CCC)— decomposed when heated in aq. soln. at 225°F. for 1 hr. Calcomine Sky Blue FF (CCC) and Calcomine Red 4BX (CCC) were stable in water alone, but showed some decomp. in the presence of Na_2CO_3 and particularly in the presence of Na_2CO_3 and cellulose. Some factors which may affect the decomp. of commercial dyes are discussed. A.J.

Principles of the Processing of Velvet Cord
N. I. Kozhin

Tekstil. prom., **17**, 28–32 (Jan. 1957)

The dyeing and finishing of velvet cord are considered critically, and special attention is paid to mercerisation, at 20–25°C., in a liquor containing 150–175 g. NaOH per litre, immediately preceding dyeing. This order of treatment gives improved mechanical properties of the velvet. G.J.K.

Oxidation Dyeing of Wool–Cotton Union Fabrics
P. P. Karpukhin and A. I. Levchenko

Tekstil. prom., **17**, 32–35 (Jan. 1957)

Wool–cotton unions are dyed in a single-bath process by impregnating the material with aniline or other aromatic amine hydrochloride or with a hydroxy compound, and oxidising with either chlorate, dichromate, or H_2O_2, the dye being produced within the fibre. Fastness to light, washing, and rubbing are satisfactory. Dyeing of wool with Aniline Black under acidic conditions requires more than twice the amount of aniline (per wt. of fibre) as compared with wool which has been previously impregnated with NaOH soln.; in the former case, the formation of Aniline Black is preceded by a chemical reaction between wool carboxyl groups and the intermediate products of the Aniline Black oxidation. G.J.K.

Application of Dyes to Acrylic Fibres
B. Kramrisch

J.S.D.C., **73**, 85–92 (March 1957)

The chief characteristics of polyacrylonitrile fibres are described briefly. Methods of application of disperse, basic (including Perkin's Mauve), Deorlene, acid, metal-complex, and chrome dyes to acrylic fibres, particularly Orlon 42 and Acrilan, are given in detail, and the main fastness properties of the dyeings are listed. Comparisons are made between the fastness properties, in particular to light and washing, of dyes on acrylic, natural, and other man-made fibres. Methods of dyeing unions of wool with Orlon 42 and Acrilan are detailed. Author

Light Fastness of Dyed Fibres— A Statistical Study
C. H. Giles

J.S.D.C., **73**, 127–160 (April 1957)

About 16,000 published commercial light-fastness assessments have been examined statistically, and from the results the following general rules are formulated, which apply to most dyes and fibres—

(*a*) On different fibres the fastness of a dye is higher the higher the regain of the fibre, provided that this is greater than about 4% (i.e. the value for nylon). Thus dyes are usually faster to light on viscose rayon than on cotton, on cotton than on nylon, on wool than on silk, and on secondary cellulose acetate than on cellulose triacetate or nylon.

(*b*) The above rule is reversed with fibres having regains below about 4%, i.e. with the newer synthetic fibres: with these the fastness is usually higher on the fibre of lower regain.

(*c*) Fastness is often reduced by (i) solid foreign materials in the fibre, e.g. dulling agents or the resin used in crease-resist finishes; or (ii) either cationic surface-active agents or powerfully hydrogen-bonding compounds, e.g. phenols or urea, used as dyeing assistants or aftertreating agents.

(*d*) The increase in light fastness with increase in depth of any dye is usually greater the higher the regain of the fibre: it tends to be higher with dyes which are insolubilised in the fibre than with water-soluble ones. The increase in average fastness for a whole class of dyes for a tenfold increase in depth is usually about 1½ units for water-soluble, disperse, and vat dyes, and about 3 units for azoic and sulphur dyes.

The effects summarised by these general principles are considered to be the result mainly of differences in the physical form of the dyes in the fibres, chemical action being important only in a few special cases, e.g. the effect of titanium dioxide delustrant. However, a more general chemical influence is the difference between the chemical nature of fading on protein and that on non-protein fibres— reduction and oxidation respectively— which has some effect on the comparative fastness of a given dye on fibres of the two classes, mainly at the upper and lower ends of the scale. Some suggestions are made of means by which light fastness might be improved, and the reliability of the fastness data is discussed. Author

PATENTS

Dye Emulsions for Dyeing and Printing Textiles
S.A.I.C.A., Societa Anonima Italiana Colori Affini

BP 768,745

Dye emulsions made with emulsifying agents which while

being soluble in both phases are more soluble in the oil than in the aqueous phase of the emulsion, e.g. the condensate of propylene oxide with an alkylamine of 8–18 C, are extremely stable and very suitable for application by the process described in *BP* 733,099 (J.S.D.C., **71**, 562 (1955)).
C.O.C.

Vatting of Vat Dyes
BASF *BP* 767,644

Complex compounds of metals which can occur in different valency states are used in a lower valency state chelated with polyhydroxyaliphatic compounds and/or hydroxycarboxylic acids as reducing agents for vat dyes. Thus $FeSO_4$ (2·5 g.) and triethanolamine (25 cc.) are dissolved in water (1 litre) and 38° Bé. NaOH (30 cc.) added. To the bath is added Indanthren Brilliant Green B (C.I. Vat Green 1) (0·6 g.). Cotton hanks treated in the resulting vat for 30 min. at 60°C. are dyed brilliant green.
C.O.C.

Dyeing Wool with Heavy Metal Dye Complexes
Gy *BP* 766,492

Wool is quickly dyed with Cr and Co complexes of monoazo and azomethin dyes (2 mol. dye to 1 atom metal) if it is first treated in dilute mineral acid so that the final pH of the liquor is 4–6 and then adding the dye to the liquor at > 80°C. in presence of capillary-active polyglycol ethers containing no nitrogen, e.g. addition product of 1 mol. 2:4-di-*tert*.amyl phenol with 12 mol. ethylene oxide.
C.O.C.

Dyeing Polyacrylonitrile Fibres
S *BP* 767,053

The material is treated with a dye liquor containing a cupric compound, the dye liquor being in contact at least intermittently during the dyeing with metallic copper. Control of the degree of reduction of the cupric compound by redox potential measurement is unnecessary; in addition level dyeing is obtained and no copper flecks can be deposited on the material being dyed. C.O.C.

Dyeing Polyacrylonitrile Fibres
Ciba *BP* 767,117

Dyeings of good fastness to light and washing are obtained by using azo dyes containing no sulphonic acid group but having at least one NH_2 group so that they form with acids salts which are at least partly hydrolysed in acid medium and fixing the dyes on the fibre as their amine salts. Thus polyacrylonitrile staple fibre is dyed with the disperse dye—

the resulting yellow dyeing is rinsed and boiled in 0·2% aq. chloroacetic acid to yield a scarlet dyeing of excellent fastness to light. Polyacrylonitrile yarn treated with salicylic acid in a boiling bath and then dyed with the above dye is also dyed scarlet of very good fastness to light. C.O.C.

Dyeing of Chrome Suède Leather
S *BP* 769,174

Wetting back can be omitted and deeper and more level dyeings obtained if the dry skins are impregnated in a drum machine with a cold concentrated solution of an acid or direct dye in presence of a wetting agent. The skins are treated until completely penetrated after which dyeing is continued with increase in the liquor ratio and finally completed at a higher temperature with a second addition of dye, applied preferably in a fresh bath. C.O.C.

Pigment–Resin Composition (IX below)
Wet Processing of Lurex (X p. 280)

IX—PRINTING

PATENTS

Pigment–Resin Composition
Rohm & Haas Co. *BP* 768,883

Dyeings or prints fast to washing and dry cleaning are obtained by using a pigment in a resinous binder containing 5–25% of a water-insoluble linear polymer of a monoethylenically unsaturated monomer containing < 1% by wt. of units of formula—

$$CH_2{:}CH{\cdot}A{\cdot}R^1OH$$

(A = O or S; R^1 = straight or branched chain alkylene of 2–10 C, $C_2H_4AC_2H_4$, $C_2H_4{\cdot}NR^2{\cdot}C_2H_4$ or $C_2H_4{\cdot}NR^2R^4X{\cdot}C_2H_4$ (R^2 = H, CH_3, C_2H_5 or C_2H_4OH; R^3 and R^4 = CH_3, C_2H_5 or C_2H_4OH; X = a negative radical)) e.g. β-hydroxethyl-vinyl sulphide or diethyleneglycol monovinyl ether.
BP 769,252

Textiles coloured with pigments carried in a resinous binder derived from copolymers containing 1–25% of a compound of formula—

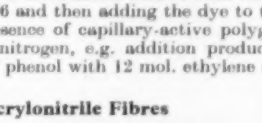
$$H_2C{:}CRCOOCH_2{\cdot}CH{-}CH_2$$
$$\qquad\qquad\qquad\quad \backslash O /$$

(R = H or CH_3) are resistant to laundering and dry cleaning.
BP 769,255

A binder comprising a water insoluble linear copolymer of monoethylenically unsaturated monomers 1–15% by wt. of which is a monomer of formula—

$$CH_2{:}CH{\cdot}O{\cdot}A{\cdot}NR^1CONHR^2$$

(A = branched or straight chain alkylene group having 2–4 C atoms between adjoining O and N atoms or ether-oxygen linked alkylene groups of 2–3 C; R^1 and R^2 = H, aliphatic of 1–3 C, or ether-linked aliphatic groups of 1–3 C, or R^1 may form with R^2 ethylene, propylene or trimethylene), e.g. $CH_2{:}CH{\cdot}O{\cdot}C_2H_4NHCONH_2$ or



is used.
BP 769,271

The binder consists of a water-insoluble linear polymer of at least one monoethylenically unsaturated monomer at least 1% by wt. being a monomer of formula—

$$CH_2{:}CR^1{\cdot}A{\cdot}R^3{\cdot}NR^4R^5$$

(R^1 = H or CH_3; A = O, COO or CO·NR^5; R^3 = straight or branched chain alkylene of 2–10 C; R^3, R^4 and R^5 = H or saturated hydrocarbon of 1–10 C), e.g. dimethylaminoethyl vinyl ether.
C.O.C.

Colouring Embossed Fabric
Rock Hill Printing & Finishing Co. *BP* 769,314

The fabric is fed through an embossing calender and while it is still on the backing roller colour is applied by a smooth surfaced roller so that only the raised portions of the fabric are coloured. C.O.C.

Light-sensitive, Heat-developable Recording Papers
M. Morrison *USP* 2,732,299

A diazotype paper is coated with a mixture of urea and gelatin. After exposure to light it may be developed by ammonia in the conventional way or by application of a non-reactive surface, e.g. Cr, at 150°C. for 1 min.
C.O.C.

Light-sensitive Pigmented or Dyed Resinous Compositions for Decorating Ceramics
Eastman Kodak Co. *USP* 2,732,297

Polymers containing units of structure—

$$R{\cdot}CR{:}CH{\cdot}CO{-}$$

(R = Alk or mononuclear Ar) are mixed with a pigment or dye, plasticiser, etc., and applied to a ceramic. The coating is then exposed to light to render only the exposed region insoluble in organic solvents, treated with an organic solvent, glazed and fired. Thus a mixture of a pigment and poly(vinylcinnamate) in ethylene glycol monomethyl ether acetate is applied to a ceramic, exposed in parts to light, treated with a mixture of xylene and ethylene glycol monomethyl ether acetate, glazed and fired. C.O.C.

Dye Emulsions for Dyeing and Printing Textiles (VIII p. 278)

X—SIZING AND FINISHING

Control of Cationic Exchange in Cotton
P. J. Hannan

Text. Research J., **26**, 948–952 (Dec. 1956)

In the impregnation of cotton fabric with copper, undesirably high uptakes of copper, due to ion exchange, may be prevented by thoroughly scouring the fabric and applying the copper in the form of a water-soluble molecular complex, e.g. with citric acid or glycine. J.C.F.

Minimum-care Cotton Fabrics
P. B. Stam, G. S. Y. Poon, M. J. Spangler, and M. P. Underwood

Text. Research J., **26**, 960–965 (Dec. 1956)

An investigation of the effects of resin type and concentration, catalyst type and concentration, and variation in fabric alkalinity in the production of minimum-care fabrics. The wide variation in results which can be realised is emphasised. J.C.F.

Minimum-care Fabrics from Viscose Rayon
T. R. Scott

Text. Research J., **26**, 966–970 (Dec. 1956)

The author discusses the requirements of minimum-care fabrics with reference to the properties of viscose rayon and the principal types of fabric treatments available. Fabrics of 100% filament viscose rayon are shown to have considerable possibilities for minimum-care fabrics. Blends with other fibres are also discussed. J.C.F.

Cellulose Interactions with certain Textile Resins
E. E. Lineken, S. M. Davis, and C. M. Jorgensen

Text. Research J., **26**, 940–947 (Dec. 1956)

The reaction of formaldehyde, melamine–formaldehyde resins, and urea–formaldehyde resins with cellulose has been studied in Cellophane films by infrared spectroscopy. The extent of reaction is shown to be directly related to the degree of crease-resistance imparted to cellulosic fabrics. J.C.F.

Swelling of Regenerated Cellulose
A. Nowakowski

J. Polymer Sci., **23**, 839–842 (Feb. 1957)

Swelling of gels of regenerated cellulose in water and organic liquids is studied, and the organic liquids are considered as means of extracting bound water. If properties of cellulose fibres are to be improved by polycondensation resins, formation of resin on the surface of the fibres must be avoided, and the resin must be incorporated inside holes in the fibre. W.R.M.

Modification of Wool with Monofunctional *iso*-Cyanates
J. E. Moore

Text. Research J., **26**, 936–939 (Dec. 1956)

The reaction of wool with (*a*) ethyl, (*b*) octadecyl, (*c*) phenyl, and (*d*) α-naphthyl *iso*cyanates in anhydrous pyridine has been investigated. At an uptake of 5% all reagents impart enhanced resistance to hot acid, the effect being least with (*a*). At higher uptakes (20–40%) (*b*), (*c*), and (*d*) reduce alkali solubility by ca. 50%. Solubility in NH_4OH after oxidation with peracetic acid is also reduced. Reaction with (*c*) and (*d*) confers resistance to shrinkage. J.C.F.

Increased Hydrophilic Behaviour of Hydroxyethylated 6-Nylon (Capron)
B. V. Petukhov and A. B. Pakshver

Colloid. J. U.S.S.R., **18**, 741–744 (Nov.–Dec. 1956)

Oriented 6-nylon fibres (in hank form) which have been treated with ethylene oxide in sealed glass tubes between room temp. and 160°C. show a considerable increase in the rate of vapour sorption and in softness, and their dyeing properties are improved. Addition of CH_3OH accelerates hydroxyethylation. G.J.K.

Wet Processing of Lurex
L. E. Seidel

Amer. Dyestuff Rep., **46**, P27–P30 (14 Jan. 1957)

Lurex is a ribbon-shaped metallic yarn in the form of a five-layer sandwich—*a*, *b*, *c*, *b*, *a*. In the centre *c* is the aluminium foil; above and below it are layers of adhesive *b*; and on the outside are layers *a* of protective plastic. The aluminium foil layer may be replaced in some forms of Lurex by finely divided metal dispersed in Dacron. The adhesive layers may contain dispersed pigment, and the outer, protective layers may be either Dacron or cellulose acetate–butyrate.

The dyeing properties of Lurexes are usually the sum of the dyeing properties of the parts, but other properties are often the result of interaction between the constituents. In boiling water Lurex may become cloudy after 8 hr., and can be cleared by heating for 3 min. at 300°F. Lurex is stable at the temperatures used in normal textile curing and drying. Dry heat, pressure, and moisture may cause separation of the layers of Lurex. Lurex is resistant to acid at moderate concentrations at low or moderate temperatures. The cellulose acetate–butyrate Lurex may be discoloured and delaminated by more drastic acid conditions. The edges of the aluminium layer are exposed in the fibre, and attack by caustic soda will dissolve the metal and cause delustring. Mercerising or vat-dyeing concentrations of alkali are usually safe, however. In general, chlorine, peroxide, and sodium chlorite bleaches do not affect Lurexes, but H_2O_2 overnight should be avoided. Dyed fabrics containing Lurex can be stripped. Soda ash will etch the aluminium in Lurex. Most dry-cleaning solvents do not affect Lurex, but acetone and trichloroethylene attack the adhesives or protective films. Vegetable and mineral oils are safe, but animal oils may delaminate butyrate Lurex. The protective films of Lurex are either cellulose acetate–butyrate or Dacron, and they dye accordingly. Thus, by choice of dye, a fabric can be dyed and the Lurex it contains left undyed, or both components can be dyed. All the usual finishing agents can be applied to Lurex. The satisfactoriness of weaving and knitting Lurex depends on tension control. If tensions in operating exceed the yield point, then the Lurex is made dull, weak, and brittle, and its chemical resistance falls.

The effects of cloth design and cloth shrinkage on the Lurex component are considered. The effects of retained alkali and retained animal oils are also considered. Precautions to be taken in wet-processing are given. In the absence of mechanical or chemical delaminating action, the light fastness of coloured Lurex is excellent. P.T.S.

Finishing Treatments for Glass Fabrics
W. Bobeth and U. Renner

Faserforsch. und Textiltech., **8**, 108–114 (March 1957)

Several methods of heat-treatment to remove softening material from glass fabrics are examined. A hot-air process at 425°C. decreases the strength of the fabric considerably, but this can be overcome by the use of certain impregnating agents. The resulting fabrics are softer and more resistant to slipping. W.R.M.

PATENTS

Antistatic Finish for Hydrophobic Synthetic Fibres
Calico Printers' Assocn. *BP* 769,014

An antistatic finish resistant to washing and dry cleaning is obtained by applying a solution of a polymer of an unsaturated monobasic acid, or a copolymer, containing unreacted carboxyl groups of one or more unsaturated mono- or dibasic acids with compounds of formula $R^1R^2C{:}CH_2$ (R^1 and R^2 = H, Ar, Alk, alkaryl, alkoxy, acyloxy, carboxy, alkoxycarboxyl or Hal) together with a polymeric polyhydric alcohol containing $\leqslant$ three ether oxygen groups per molecule, and then baked. Thus Terylene is treated with an alkaline aqueous solution of poly(methacrylic acid) and a polyethylene glycol of mol. wt. 600, dried and baked for 1 hr. at 130°C. C.O.C.

Permanent Finish
FH *BP* 766,330

Fabric is woven from a warp sized with an aqueous dispersion of a vinyl polymer and a size which is either soluble or swells in water. The resulting fabric is hot calendered. The finish is faster to washing than that obtained by impregnating the cloth with the same agents. Thus a cotton warp is sized with farina (25 kg. per 500 l.), pearl glue (2) and 50% aq. dispersion of polyvinyl acetate (40). It is woven up with unsized weft and the cloth steamed and calendered at 90–120°C. It has a full handle which is maintained even after several boiling washes. Treating a woven fabric with the size gives the same full handle but it becomes flabby after the first wash. C.O.C.

Chemical Modification of Cellulose
Fothergill & Harvey (Research & Development) *BP* 767,991

Monoglycidyl ethers are equally as effective as the bisglycidyl ethers described in *BP* 757,386 (J.S.D.C., **72**, 615 (1956).) for introducing groups into the cellulose molecule although unlike the bisglycidyl ethers they do not cause cross linking. Thus viscose rayon cloth was treated in 18% NaOH and then heated at 100°C. for 20–35 min. in a 33% solution of the monoglycidyl ether of phloroglucinol in 50% aqueous dioxane. It was then washed thoroughly

in dil. H_2SO_4, in boiling dioxane, in hot soap solution and finally in boiling water. It was finally dried and treated with Brentamine Fast Red B Salt (C.I. Azoic Diazo Component 5). The resulting dyeing was unaffected by boiling acetone, 1% soap solution or hot water.

C.O.C.

Treating Cellulose with β-Propiolactone or β-*iso*Valerolactone

U.S. Secretary of Agriculture *USP* 2,731,323

The fibres are impregnated with 5–50% of aq. alkali metal hydroxide and then treated at 0–30°c. with the lactone either in the pure state or as a solution of < 5% strength until they contain 0·5–26% of their weight of products of the reaction. C.O.C.

Imparting Hydrophilic Properties to Cellulose

DuP *USP* 2,732,317

The cellulose, in any desired form, is immersed in a solution of a polymerisable vinylidene monomer in presence of a quaternary ammonium salt whose anion has mol. wt. < 150 and of an initiator for the addition polymerisation of the monomer. After polymerisation is complete the material is thoroughly washed with a solvent to remove all surface polymer. Thus viscose rayon yarn is wet out and then immersed for 1 hr. in an aqueous solution of β-methacryloxyethyltrimethylammonium methosulphate for 1 hr. in presence of *aa'-iso*bis(*iso*butyramidine hydrochloride). The yarn is taken out, drained, heated in a closed container at 70°c. for 16 hr. and treated in boiling water for 1 hr. to remove surface polymer. The resulting semiglutinous yarn is treated with methanol and dried at 70°c. under reduced pressure. The treated yarn is double the weight of the untreated yarn and has regain of 75·8% at 97% R.H. compared with 38·9% for the untreated yarn. C.O.C.

Improving the Tensile Strength of Cotton

Institute of Textile Technology *BP* 767,810

Cotton as loose fibres, yarn or fabric is treated with 3-*iso*propoxy-, 3-methoxy-, 3:3'-aminobis-, 3-dimethylamino- or 3-*iso*propylamino-propylamine while in liquid phase (including aqueous solution) at normal room temperature and pressure until the swelling action is completed. The treated fibres show decrease in crystallinity and increase in tensile strength, elongation and abrasion resistance.

BP 767,811

Cotton or partly cyanoethylated cotton is similarly treated but under tension with methylamine, ethylamine, di- or trimethylamine, triethylamine, triethanolamine, ethylene diamine or diethylene triamine, or in the case of cyanoethylated cotton with the amines mentioned in the previous abstract. The treated material is washed while still under tension and the tension then released.

C.O.C.

Fixing Mechanical Effects and Finishes on Textiles containing Regenerated Cellulose

British Industrial Plastics *BP* 767,789

The materials are impregnated with an aqueous solution containing a lower alkyl ether of dimethylol urea (75% of its methylol groups etherified), and an acid or potentially acid catalyst, at least partly dried, given the desired mechanical finish, and baked. C.O.C.

Coating Cotton with Dextran to render it Resistant to Chemicals

Commonwealth Engineering Co. *USP* 2,731,369

The cotton is treated with a solution of dextran, then treated with an alkylating agent in alkaline solution and finally baked. Thus cotton is treated with dextran dissolved in aq. NaOH, treated with diethyl sulphate dissolved in aq. NaOH and baked at 100–115°c.

C.O.C.

Imparting Increased Elasticity to Woven and Knitted Fabrics

K. Wirz *BP* 767,287

The fabric in relaxed condition is treated with a mixture of a linear polyester and a di- or poly*iso*cyanate. The polyester has at least at both ends hydroxy or carboxyl groups and is at a low mol. wt. degree of polymerisation. The mixture is applied from organic solution. Final polymerisation is effected on the fibre by heating for several hours at 80–120°c. Thus knitted rayon fabric is impregnated with a 10–20% solution in anhydrous *cyclo*hexanone of an equimolecular mixture of hexamethylene di*iso*cyanate and a polyester of adipic acid and 1:2-ethanediol having terminal hydroxyl groups and mol. wt. about 1000. It is dried at 110°c. and kept at that temperature until the reactants have completely interlinked both with themselves and with the fibres. C.O.C.

Rendering Textiles Resistant to Shrinking and Retention of Creases

American Cyanamid Co. *USP* 2,730,427

Textiles containing functional groups, e.g. wool, cotton, viscose rayon, are treated with 2–20% of their dry weight of a compound of formula—

$$H_2C\!-\!CHCH_2\cdot O\cdot R\cdot O\cdot CH_2CH\!-\!CH_2$$
$$\quad\backslash O/\qquad\qquad\qquad\quad\backslash O/$$

(R = alkylene of 2–6 C) dried and baked. Thus cotton cloth is impregnated with a 10% aq. soln. of ethylene glycol bisglycidyl ether containing 1% of a mixture of diammonium phosphate (30) and hexamethylene tetramine (4) so that on drying it contains 6·2% of the ether. After drying it is baked at 350°*F.* for 10 min. After being washed for 1 hr. the treated cloth has a warp shrinkage of 3·1%, whereas the untreated cloth shrank 6·7%. C.O.C.

Mothproofing with Soluble Dicyandiamides

American Cyanamid Co. *USP* 2,732,277

Treatment with a hot or cold aqueous solution of a dicyandiamide salt, if desired simultaneously with another aqueous treatment, e.g. dyeing, and then rinsing or soaping, imparts a mothproof finish of good fastness to light, washing, dry cleaning, and storing. Even when the amount of dicyandiamide salt left on the wool after rinsing is so small as to be undetectable by normal methods of nitrogen analysis the wool is just as repellent to moth grubs as wool containing much more of the dicyandiamide salt. Thus wool impregnated with 4% of its dry weight of ammonium dicyandiamide in aqueous solution and dried and then washed and dry cleaned several times was mothproof.

C.O.C.

Treating Acetate Rayon with Elastomers

Celanese Corpn. of America *USP* 2,731,368

Acetate rayon cloth, or cloth made from other fibres made from organic derivatives of cellulose, when treated with an elastomer, e.g. poly(vinyl butyral), vinyl chloride-vinylidene chloride or butadiene-styrene copolymers is given increased recovery from creasing and a firmer, fuller handle without any tendency to stiffness. Thus acetate rayon cloth impregnated with 0·625% of its dry weight of a mixture of 20% by wt. poly(vinyl butyral) and 80% butyl ricinoleate had a recovery of 134° after 5 min. whereas an untreated sample had a recovery of only 98°, both cloths being creased by being folded and placed for 5 min. under a 1000 g. weight after being conditioned for 24 hr. at 24°c. and 65% R.H. C.O.C.

Curing of Resin-treated Fabrics, etc.

V. E. Yarsley, W. Flavell and B. Frenkel *BP* 767,502

The resin treated material is cured by passing it through a hot liquid in which it floats but is prevented from breaking the surface by plates placed below the surface of the liquor. The liquid must be inert to the materials being treated. Passage through the liquor also provides the pressure needed in the case of laminates to bring the components into sufficiently close contact, the pressure being provided either by static head or by hydraulic pumps which circulate the liquid. Thus glass cloth treated with a polyester resin can be passed through water at 90°c.

C.O.C.

Water-repellent and Crease-resistant Finish

ICI *BP* 767,834

A combined water-repellent and crease-shedding finish is obtained by adding to the precondensate impregnating liquor a Werner type complex chromium compound. Thus spun viscose fabric is impregnated with its own wt. of the solution made by mixing 37 parts of urea-formaldehyde precondensate (32° Tw.), 3 parts of a 30% solution of stearato chromic chloride in *iso*propanol and 60 parts of water. The impregnated fabric is dried at 60°c. and baked for 3 min. at 130°c. The resulting water-repellent and crease-shedding finish is durable to washing and dry cleaning. C.O.C.

Water-repellent Finish
FBy *BP* 767,945

The goods are impregnated with an aqueous solution or dispersion of a salt of a carbamic acid whose N atom carries both a H atom and an aliphatic hydrocarbon radical having a chain of > 7 C which may be interrupted by hetero atoms with an amine derived from carbamic acid by loss of CO_2, e.g. the stearylamine salt of stearylcarbamic acid, and dried at > 100°C. Thus the adduct of $NaHSO_3$ and stearyl *iso*cyanate (25 g.) is dissolved in 500 cc. water at 85°C. After 10 min., 100 cc. of water at 30°C. are added and at 70°C. 52 cc. of *N*-NaOH and finally after 10 min. the volume is brought up to 1 litre with water at 60°C. Cotton gaberdine is impregnated with 60% of its weight of this solution, dried at 100°C. and baked for 5 min. at 120°C. The excellent water-repellent finish obtained resists repeated dry cleaning and the fabric has a soft handle. C.O.C.

Water-repellent Finish
ICI *BP* 763,464

Textiles are treated with a silicone oil emulsified in an aqueous solution of an alkali metal salt of an alkyl, aryl or aralkyl silicone acid and a water-soluble thermosetting synthetic resin precondensate at pH 2–8 and then dried and baked. C.O.C.

Water-repellent Finish
FH *BP* 767,585

A good hydrophobic effect and excellent capacity for repelling water droplets are obtained by treating fibrous materials with a solution in an inert organic solvent of (1) a reaction product of an aluminium alcoholate (1 mol.) with a monobasic organic substance (0·2–2·5 mol.) capable of forming an aluminium salt, and (2) a paraffin hydrocarbon of > 15 C and/or a natural or synthetic wax consisting of compounds of high mol. wt. linked in an ester-like manner and/or a viscous to solid polychlorinated hydrocarbon of > 9 C, and then drying at > 50°C. preferably in presence of moisture. Thus cotton poplin is treated for 5–10 min. at 60°C. in a dry cleaning machine and a goods : liquor ratio of 1 : 10 with carbon tetrachloride containing per litre 10 g. of the condensate of 1 mol. of Al *iso*propylate with 0·65 mol. of stearic acid and 10 g. of paraffin wax. The fabric is centrifuged and dried for 15 min. at 60°C. with air at 30% R.H. An excellent water-repellent effect is obtained. C.O.C.

Giving Nylon Stability and Resistance to Solvents
General Electric Co. *BP* 767,203

Irradiating nylon with high energy electrons enhances its resistance to solvents and reduces its susceptibility to plastic flow. C.O.C.

Improving the Pilling Resistance of Cloths containing Nylon, Polyester or Polyacrylic Fibres
Fontaine Converting Works *BP* 766,384

The fabric is treated at ≤ 350°F. with a liquid of b.p. above the temperature of treatment, washed free of the liquid and dried. Thus Terylene fabric is passed through diethylene glycol at 360–365°F. for 10 sec., rinsed with water and dried. C.O.C.

Durable Plastic Floor Coverings
Armstrong Cork Co. *USP* 2,731,358

The floor coverings obtained by printing a vinyl resin composition onto a coated felt base are not subject to gouging (marking when furniture is moved on them) if 3–20% of the solids in the coating paint binder is casein. C.O.C.

Flexible, Porous, Metallised Sheet Material
ICI *BP* 767,714

Cloth, paper, non-woven fibrous web, etc., are sprayed or coated with an adhesive in such a manner, e.g. by a tipping roller, that the treated material is permeable to air. A thin porous layer of metal is then applied on top of the adhesive by either a transfer method or by vacuum deposition. C.O.C.

Coated Non-woven Fibrous Sheets
Johnson & Johnson (Gt. Britain) *BP* 763,456

A layer of thermoplastic film-forming material is applied to a layer of non-woven oriented fibres bonded together at points the distance between which is less than that of the average fibre length. The material is applied so that most of the fibres project from one surface only of the product. C.O.C.

Coated Webs
Kimberley-Clark Corpn. *BP* 763,453

A web composed of filaments is coated with a dispersion of viscosity 2,900–16,000 centipoises and containing a thermoplastic water-insensitive resin and a plasticiser. The coated web is then heated so as to produce a dry surface coating without the resin penetrating the filaments. The product is soft, flexible and unaffected by water. C.O.C.

Degradation during Storage of Viscose Rayon Staple (VI p. 276)
Principles of the Processing of Velvet Cord (VIII p. 278)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Infrared Absorption Spectra of Mono- and Polysaccharides
A. A. Konkin, D. N. Shigorin, and L. I. Novikova
Faserforsch. und Textiltech., **8**, 85–90 (March 1957)

Infrared absorption spectra for monosaccharides, polyhydric alcohols, and polysaccharides suggest the existence of five possible hydrogen bonds. The energies of these are calculated, and their contributions to the total interaction due to hydrogen bonds in polysaccharides estimated. W.R.M.

Cellulose, Starch, and *cyclo*Glucans
K. Freudenberg
J. Polymer Sci., **23**, 791–799 (Feb. 1957)

A review of the kinetic and preparative evidence for the structure of cellulose, starch, and *cyclo*glucans. W.R.M.

Sorption of Iodine and Crystallinity of Cellulose Fibres. XIII—Colour and Causes of Iodine Sorption on Cellulose and Cellulose Acetate Fibres
K. Schwertassek
Faserforsch. und Textiltech., **8**, 65–69 (Feb. 1957)

Reply to criticism by T. G. Majury (*Text. Research J.*, **26**, 437 (1956)). Crystallisability of iodine compounds with cellulose seems to be related to the colour produced, poor crystallisability being indicated by a brown colour and good crystallisability by a blue. Crystalline cellulose seems to adsorb iodine with a slightly blue or violet colour. Different colours may result from carrying out treatment of cellulose with iodine under different conditions. The nature of the combination of iodine with cellulose and cellulose acetate is discussed. W.R.M.

Molecular Fractionation of Cellulose
H. Sihtola, E. Kaila, and L. Laaman
J. Polymer Sci., **23**, 809–824 (Feb. 1957)

Fractionation based on differential dissolving power of cupriethylenediamine or cuprammonium hydroxide solutions containing different concentrations of copper does not seem to be possible. Addition of acid to cupriethylenediamine solution does not result in fractionation according to chain length, which does occur if organic liquids, such as propanol, are used as precipitants. Since depolymerisation occurs in cupriethylenediamine and cuprammonium hydroxide solutions, these solvents are not suitable for chain-length determination. W.R.M.

Chemistry of Cuprammonium Solutions of Cellulose. VII—Interaction of Polyhydroxy Compounds with Cuprammonium Complexes
S. N. Danilov and M. G. Okun'
J. Gen. Chem. U.S.S.R., **26**, 3005–3014 (Nov. 1956)

Analysis of precipitates formed on addition of NaOH and LiCl to cuprammonium solutions of cellulose show a variable composition in presence of large concn. of caustic (cellulose : Cu ratio approx. 1 : 1). The absorption spectra of cuprammonium solutions of cellulose and of other low- and high-molecular polyhydroxy compounds are shown not to differ from those of Schweizer's reagent itself, and it is concluded that no characteristic compounds between cuprammonium complexes and cellulose exist, but colloidal phenomena, with probably the formation of very unstable molecular compounds, are suggested. G.J.K.

Rôle of Phosphoric Acid in the Study of Cellulose I—Swelling and Dissolution of Cellulose in Ortho-phosphoric Acid
S. N. Danilov and N. F. Gintse
J. Gen. Chem. U.S.S.R., **26**, 3014–3020 (Nov. 1956)
Solubility studies of cellulose (linters) in H_3PO_4 of different concn. underline the fundamental rôle of hydrates in the dissolution of cellulose in conc. soln. of electrolytes. Changes in η of cellulosic soln. in conc. H_3PO_4 occur on heating (above 35°c.) or on prolonged standing. G.J.K.

Cellulose–Methanol System
K. Lauer, J. E. Ayer, and A. L. A. Seoud
J. Polymer Sci., **24**, 67–74 (March 1957)
Determination of non-solvent methanol in cotton and mercerised cotton fibres and comparison with values for non-solvent water show that methanol solvates only half the hydroxyl groups available for hydrate formation in amorphous regions. A schematic approach to fibre structure gives good agreement between the quantities of non-solvent water and methanol and molar integral heats of swelling respectively. Both characteristics permit estimation of fibre composition. W.R.M.

Cellulose Derivative–Solvent Interaction
W. R. Moore, J. A. Epstein, A. M. Brown, and B. M. Tidswell
J. Polymer Sci., **23**, 23–46 (Jan. 1957)
Osmotic pressure, precipitation, and viscosity results are given for dilute solutions of cellulose nitrate, cellulose acetate, and ethylcellulose of similar molecular weight and degree of substitution in a fairly wide range of solvents. Values of the Huggins interaction parameter μ are obtained, and an attempt is made to interpret their variation with solvent. Criteria for the use of the dilution ratio method in the estimation of solvent power are suggested. Values of suggested measures of solvent power are obtained from viscosity measurements and compared with values of μ. In certain cases both the intrinsic viscosity and the initial slope of the viscosity number–concentration plot increase with solvent power. Other suggested measures, applicable to systems containing flexible polymers, do not seem to apply to cellulose derivatives. W.R.M.

Relative Reactivity of Hydroxyl Groups of Cellulose in Methylation
Z. Rogovin and V. Derevitskaya
Faserforsch. und Textiltech., **8**, 61–64 (Feb. 1957)
Determination of the position of methyl groups in partly methylated celluloses shows the secondary hydroxyl groups to be more reactive than the primary. W.R.M.

Relative Reactivity of the Hydroxyl Groups in Cellulose
V. Derevitskaya, Yu. Kozlova, and Z. Rogovin

II—Distribution of Methoxyl Groups in partly Methylated Cellulose obtained in Basic Media
J. Gen. Chem. U.S.S.R., **26**, 3369–3374 (Dec. 1956)
A partial methylation of cellulose is achieved by the use of CH_3I and $(CH_3)_2SO_4$ in presence of benzyltriethyl-ammonium hydroxide (case I) and also by methylating a cellulose copper–sodium compound (case II). In I max. reactivity is shown by secondary hydroxyl groups, in methylcellulose with $\gamma \simeq 100$ the ratio to primary hydroxyl being 2 : 1. In II for $\gamma \simeq 100$ reaction takes place almost exclusively on the secondary hydroxyl groups, the presence of methoxyl groups preferentially attached to one of the secondary carbon atoms being confirmed by HIO_3 oxidation of non-methylated glycollic groups. With methyl-cellulose in presence of base the distribution of methoxyl groups depends on the nature of the methylating agent; similarly, change of base gives a different distribution of methoxyl groups, methylation taking place at the expense of hydroxyl groups which had undergone reaction with the base.

III—Distribution of Methoxyl Groups in partly Methylated Cellulose obtained with Diazomethane as Methylating Agent
Ibid., 3374–3376
Results found in Part II are confirmed when cellulose methylated with diazomethane is investigated. G.J.K.

Preparation and Properties of Low-substituted Methyl- and Carboxymethyl-cellulose
N. I. Nikitin and G. A. Petropavlovskii
J. Appl. Chem. U.S.S.R., **29**, 1540–1549 (Oct. 1956)
Methylcellulose is prepared by the action of dimethyl sulphate on cotton, previously treated in 12–18% NaOH soln. for 1 hr. The methylation is carried out at 15°c. for 4 hr. in ether soln. The action of monochloroacetic acid on cotton in 18% excess of NaOH results in the formation of a carboxymethyl derivative. The reaction is carried out at 40°c. and is completed after 4 hr. Both derivatives are soluble in 4–6% aq. NaOH soln. after they have been subjected to a temp. of − 15°c. T.Z.W.

Structure of Highly Substituted Hydroxyethyl-celluloses
O. M. Klimova and I. A. Katsizno
J. Appl. Chem. U.S.S.R., **29**, 1550–1555 (Oct. 1956)
Hydroxyethyl ethers of cellulose containing 1–2·5 hydroxyethyl groups are prepared by the action of ethylene oxide on cellulose mercerised for 24 hr. at 18°c., hydroextracted to 3 times its own weight, and ripened at room temp. for 40 hr. The reaction is carried out in an autoclave at 18–20°c. for 30 hr. The proportions of ethylene oxide used varied from 11 to 33 mol. per mol. of cellulose. The reaction is one of combination of ethylene oxide with primary alcoholic groups of cellulose, and only a very small proportion of the oxide combines with secondary alcoholic groups. When too great an excess of ethylene oxide is used, polyethylene chains are formed, which affect negatively the reactivity to a second etherification. T.Z.W.

Solvation of Cellulose Ethers in Mixed Solvents
S. A. Glikman and L. A. Root
Colloid J. U.S.S.R., **18**, 656–659 (Nov.–Dec. 1956)

Change of Double Refraction of Cellulose Triacetate on Heating
A. Möhring and G. Duwe
Faserforsch. und Textiltech., **8**, 54–61 (Feb. 1957)
Acetylated natural fibres, wet-spun cellulose triacetate fibres, drawn foils, and bristles show a marked change of double refraction at 190–200°c. on heating or cooling. Double refraction decreases during heating and may temporarily change sign, but after cooling it is greater than before heating. Such variations are attributed to changes in crystalline structure. W.R.M.

PATENTS

Improving the Consistency of Paper Stock
Monsanto *BP* 765,727
The consistency of paper stock can be improved without appreciable increase in viscosity by adding 1–10% by weight (based on the solids content of the slurry) of a free water-soluble, molecularly dehydrated inorganic hexametaphosphate salt having a ratio of $Na_2O/P_2O_5 < 1{\cdot}7$. K.W.

Beater Sizing of Paper with Ketene Dimers
Hercules Powder Co. *BP* 765,715
An aq. suspension of a ketene dimer, $(RCH:CO)_2$ (R = Alk of ≥ 8 C atoms, *cyclo*alkyl of ≥ 6 C, Ar, aralkyl, or alkaryl group), e.g. hexadecyl ketene dimer, is added at pH 5–9 in concn. of 5–200 p.p.m. water to paper stock during stock preparation. K.W.

Mineral Coated Paper
S. D. Warren *BP* 769,460
In the manufacture of cast-coated paper, oily release agents, which ensure separation of the coated surface from the casting surface, are replaced by an aq. solution or dispersion containing 0·25–25% of an alkali resinate or ammonium resinate. K.W.

Improved Dry Strength of Paper
American Cyanamid *BP* 769,732
An aq. suspension of a linear polyalkane polymer or a linear polyaza-alkane polymer, containing 1–15 quaternary ammonium groups, and having an average mol. wt. of 50,000–200,000, is added to a pulp suspension containing 2–30% of a carbonate filler. K.W.

Fibrous Products having Increased Tear Strength
Rohm and Haas Co. *BP* 764,147
An aq. fibre suspension is pretreated with a poly-cationic N compound, e.g. poly-quaternary NH_3 compounds

having preferably mol. wt. 600–3000, e.g. condensate of triethylenetetramine with 1·6 moles. of ethylene dichloride, followed by addition of an aq. dispersion of one or more synthetic linear polymers of vinylidene compounds containing COOH-groups, e.g. acrylic acid polymers having mol. wt. 1000–300,000. Non-ionic dispersing agents may also be added to the suspension which has a pH 7–9·5. The amount of vinylidene polymerised is 10–50% on the weight of the fibre. K.W.

Photographic Light-sensitive Paper
Kodak *BP* 766,979

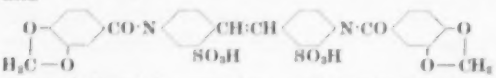
Base paper is coated with pigment ($BaSO_4$) suspended in an aq. suspension of a colloidal binder consisting of at least 25% styrene-butadiene resin, with or without the addition of gelatine, in a pigment-binder ratio of between 1 : 1 and 10 : 1, and then heat-treated at 275–375°F. An intermediate layer of styrene-butadiene resin followed by a layer of light-sensitive gelatino-silver halide emulsion are applied subsequently to the coating. K.W.

Imparting a Hard Finish to Waxed Paper
Charlestown Paper Converting Co. *BP* 768,565

Waxed paper is led, whilst the wax is still unsolidified, into a tank in which a stream of water at 32–46°F. impinges tangentially on each side of the web at a velocity approx. one third that of the web. A hard finish is thereby given to the wax coating. K.W.

Colour-stabilised White-pigmented Ethyl Cellulose Plastic Compositions
Dow Chemical Co. *USP* 2,731,357

Incorporation of 0·2 p.p.m. to 10% by wt. of 4:4′-bis(3:4-methylenedioxybenzamido)-stilbene disulphonic acid



or an alkali metal, ammonium or alkylamine salt thereof gives good colour-stabilisation of white-pigmented ethyl cellulose composition to prolonged exposure to heat. C.O.C.

Light-sensitive, Heat-developable Recording Papers (IX p. 279)
Swelling of Regenerated Cellulose (X p. 280)
Flexible, Porous, Metallised Sheet Material (X p. 282)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Potentiation of Chemical Reactivities at Protein Surfaces
H. P. Burchfield
Nature, **179**, 630 (23 March 1957)

Conditions leading to suppression or increased ionisation of functional groups may lead to increased reactivity. Two ways by which this may occur are suggested. The first entails isolation of functional groups from surrounding carboxyl groups. Thus, asparagine is 6 times as reactive as aspartic acid, which indicates that the second carboxyl group has suppressed reactivity by increasing the pK' value of the amino group. The second mechanism is one which reduces pK' values to 7 or less in order to utilise maximum latent reactivity. It is believed that this can be achieved if the ionising group is situated in a region rich in positively charged centres with high pK' values such as the guanido groups of the arginine residue. These would tend to repel protons and attract negative ions, creating a micro-environment with a higher pH than the main body of the system. A larger proportion of the functional groups would then ionise. W.R.M.

Swelling of Fibrous Proteins
J. L. Morrison and J. F. Haslam
Nature, **179**, 528–529 (9 Mar. 1957)

Heats of wetting are combined with water-vapour adsorption isotherms to obtain thermodynamic properties. Variations of the differential free energies, heats, and entropies are illustrated. It is suggested that at low regains the material is disorganised, and water vapour is bound by hydrogen bonds to give a monolayer. Ordering due to adsorption is greater than disordering due to swelling. After completion of the monolayer a second layer begins to build up, and the degree of ordering falls off. Below 20% regain the water is compressed or fills molecular interstices. Above 20% regain intermicellar rather than intermolecular swelling occurs. W.R.M.

XIII— RUBBER; RESINS; PLASTICS

Interaction of 6,6-Nylon with Solvents
L. Valentine
J. Polymer Sci., **23**, 297–312 (Jan. 1957)

Swelling of cross-linked 6,6-nylon is interpreted on the basis of the Flory–Rehner treatment of swelling. Values of the polymer–solvent interaction parameter χ are obtained and discussed in terms of properties of the swelling liquids. The solvent power of about twenty phenols can be correlated with their ability to form hydrogen bonds. Intramolecular bonding reduces solvent power. Some implications in use of the solvents as plasticisers and dyeing assistants are suggested. W.R.M.

Crystallisation of 6,6-Nylon
E. H. Boasson and J. M. Woestenek
J. Polymer Sci., **24**, 57–61 (March 1957)

A study of crystallisation in 6,6-nylon on cooling after fusion at temperatures just above the m.p. Optically negative spherulites are obtained by crystallising at temp. of 250–258°C. These have a higher optical m.p. than optically positive spherulites previously observed in 6,6-nylon. Another type of spherulite is reported. W.R.M.

Acrylonitrile Polymerisation
W. M. Thomas, E. H. Gleason, and G. Mino
J. Polymer Sci., **24**, 43–56 (March 1957)

Polymerisation of acrylonitrile in dilute aqueous solution initiated by the chlorate–sulphite redox system is studied kinetically and by the electron microscope. The rate of polymerisation depends on the product of the concentrations of chlorate and sulphite, the monomer content, and the concentration of sodium lauryl sulphate. The rate is decreased by agitation and salts but accelerated by Fe^{2+} and Cu^{2+}. Particles of polymer separate from the start as spheroids. The system bears a general resemblance to typical emulsion polymerisation. W.R.M.

Structure and Properties of Long-chain Polymers in Dilute Solutions. I— Solutions of Polyvinyl Chloride and Polyacrylonitrile
N. V. Mikhailov and S. G. Zelikman
Colloid J. U.S.S.R., **18**, 717–723 (Nov.–Dec. 1956)

Glass Temperatures of Polychlorotrifluoroethylene, Polyvinylidene Fluoride, and their Copolymers
L. Mandelkern, G. M. Martin, and F. A. Quinn
Bur. Stand. J. Res., **58**, 137–143 (March 1957)

The glass temperature of polychlorotrifluoroethylene is found to be 45°C. An upper temperature for that of polyvinylidene fluoride is − 35°C. Glass temperatures for copolymers are between the values for the homopolymers. Results are in agreement with theoretical deductions relating the glass temperature of a copolymer to its composition. W.R.M.

Flexible, Porous, Metallised Sheet Material (X p. 282)

XIV— ANALYSIS; TESTING; APPARATUS

Evaluation of the Usefulness of Conditioning Ovens in relation to the Correctness of their Working (I p. 269)
Molecular Fractionation of Cellulose (XI p. 282)
Chemistry of Cuprammonium Solutions of Cellulose. VII— Interaction of Polyhydroxy Compounds with Cuprammonium Complexes (XI p. 282)

XV— MISCELLANEOUS

Modern Methods of Research
L. A. Jordan
Chem. and Ind., 306–318 (16 March 1957)

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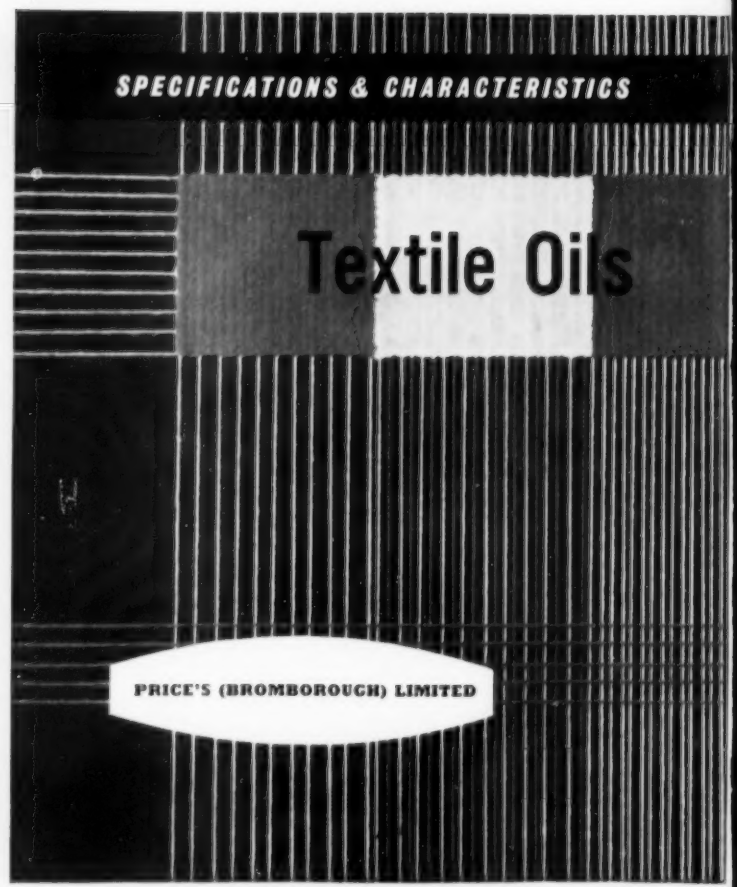

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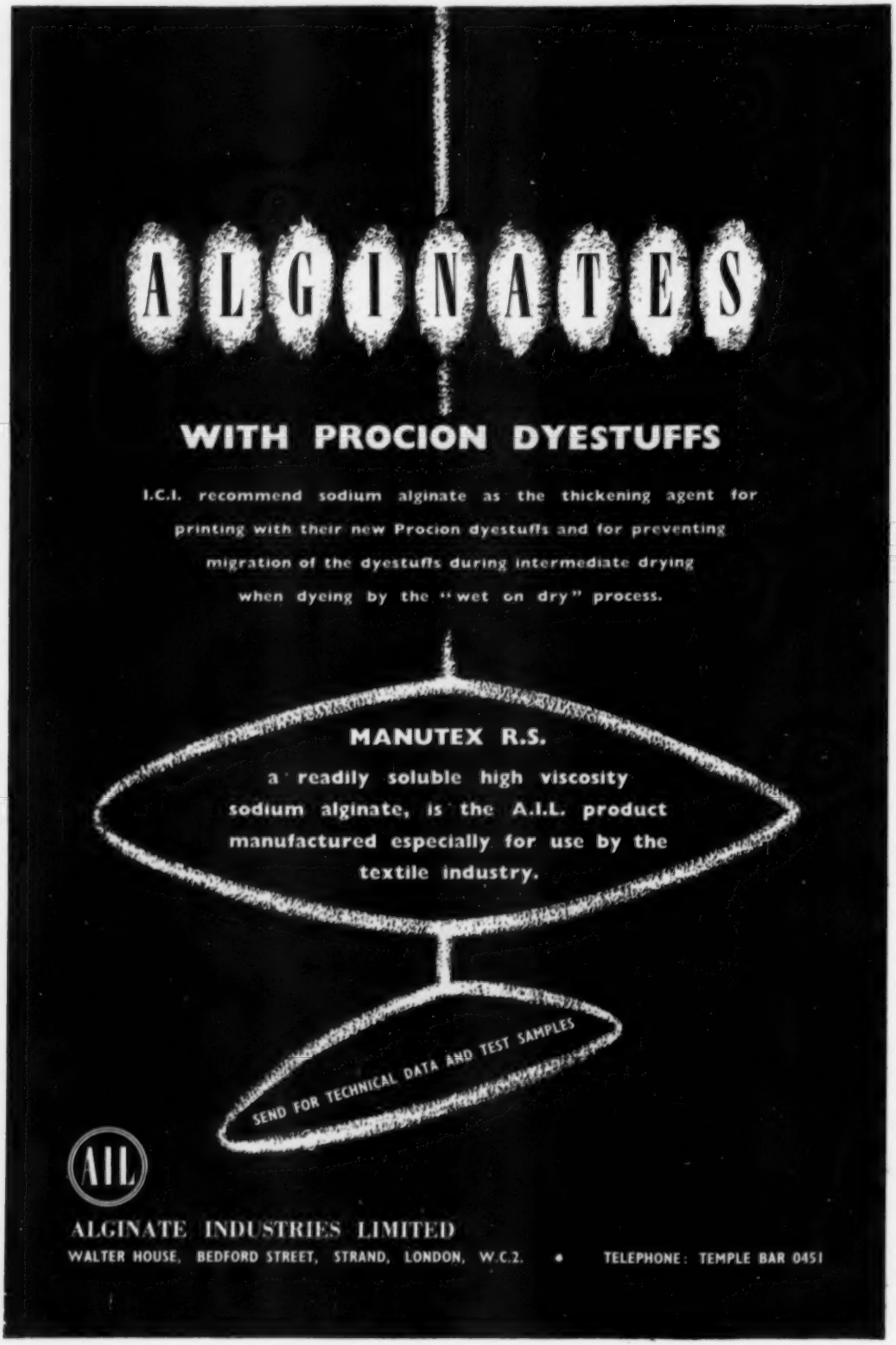
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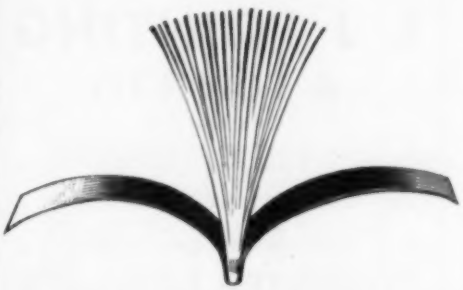

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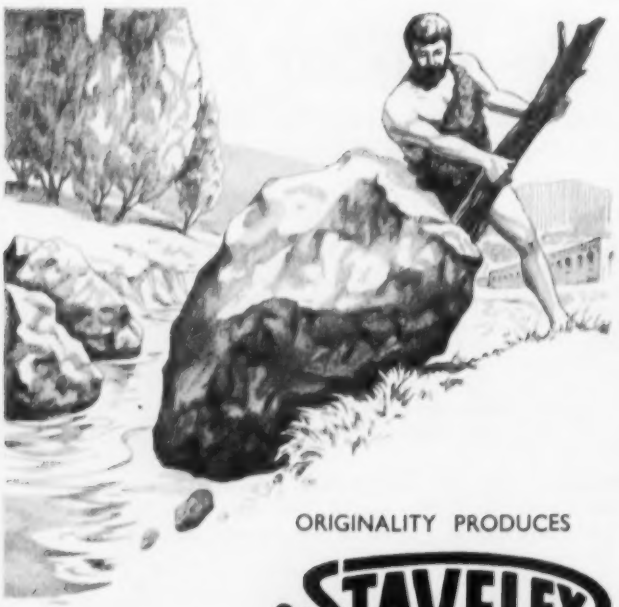

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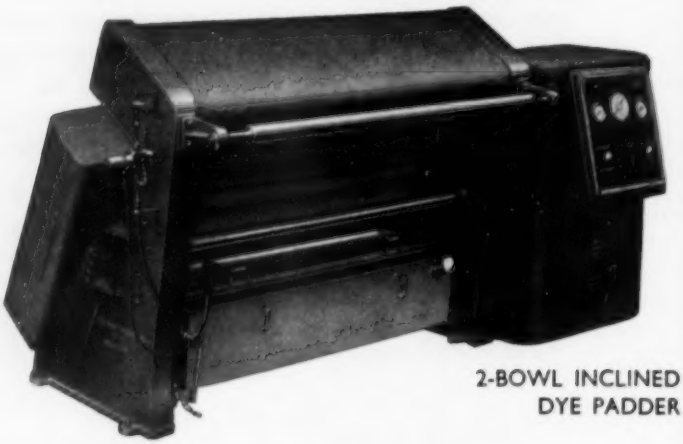

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